LIBSCAN50™ & LIBSCAN100™

Modular LIBS System

User's Manual

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APPLIED PHOTONICS

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1 Introduction

The ***LIBSCAN Modular LIBS System*** is designed to be a highly versatile, adaptable and upgradeable product suitable for use either in a laboratory environment or in the field. The modular design is also particularly well-suited to research and development work and hence LIBSCAN will be of value to organisations who wish to conduct their own LIBS R&D program and who may wish, for example, to integrate LIBSCAN with an industrial process control application. LIBSCAN is currently available with either a 50 mJ 1064 nm laser (LIBSCAN 50) or a 100 mJ 1064 nm laser (LIBSCAN 100), although versions configured for alternative laser wavelengths (355 nm, 266 nm) and with double-pulse option are available as a special order. LIBSCAN is available with either six or eight plasma light collection channels and so therefore may be used with up to eight individual spectrometers. Various types of modular sample chamber are available for LIBSCAN including one equipped with a computer-controlled, multi-axis sample stage.

This User's Manual provides the necessary information needed to configure the integrated LIBSCAN system and how to use it safely. It is assumed that the user will be familiar with the safety issues arising from the use of high-power lasers and will have preferably been trained in the safe use of laser products.

Examples of typical configurations of the LIBSCAN system, including illustrations of how it may be used with various sample chambers, are given in Appendix A2.

2 Safety

2.1 Laser radiation

LIBSCAN contains a high-power Q-switched Nd:YAG laser (Class 4) and so it is imperative that it is operated only by suitably trained and experienced persons who are fully aware of the hazards inherent to this type of high-power laser equipment. It is imperative also that, prior to using the equipment, an appropriate risk assessment is conducted in such a way as to take account of the proposed use of the equipment, the environment in which the equipment is to be operated, and how its use may affect people who are not directly involved with the use of the equipment.

WARNING
CLASS 4 LASER PRODUCT
VISIBLE AND INVISIBLE LASER RADIATION
AVOID EYE OR SKIN EXPOSURE TO DIRECT OR SCATTERED RADIATION
Wavelength: 1064 nm
Pulse repetition rate: 20 Hz
Pulse length: 3 - 10 ns
Pulse energy (max.): 100 mJ
Peak power (max.): 35 MW
Average power (max.): 2 W

LIBSCAN is designed to meet the laser safety requirements of the relevant European standards (BS EN 60825) and USA standards (ANSI Z136.1 – 2007). Although LIBSCAN may be supplied with a sample chamber which provides adequate containment of the laser radiation to Class 1 Accessible Emission Limits, it is possible for the user to operate LIBSCAN without the sample chamber (ie. operation in "open beam" mode as may be required for certain types of experiment). Accordingly, it is necessary to consider LIBSCAN to be a **Class 4 Laser Product** and so, by definition, the equipment poses a risk of personal injury (eye, skin injury) and poses a fire risk. As with all Class 4 laser products, appropriate safety precautions must be taken as identified via a suitable risk assessment conducted by the user in consultation with a suitably qualified and experienced Laser Safety Officer.

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The most significant hazard relating to exposure of personnel to the laser radiation is eye injury since direct or scattered laser radiation produced by LIBSCAN can cause serious and permanent injury to the eyes including blindness - such injury may be instantaneous. Precautions must be taken to avoid exposure of personnel to hazardous levels of laser radiation. Such precautions may include the setting up of a temporary or permanent laser controlled area (eg. a laser laboratory). Other measures may also be necessary, as determined by appropriate and thorough assessment of the risks (ie. a risk assessment) conducted by the personnel responsible for the safe use of the LIBSCAN system. Consult the manual supplied with the laser for further guidance on the safe use of the laser.

The door of the sample chamber is equipped with a dual electrical interlock switch which is designed to prevent activation of the laser unless the door is fully closed. The door interlock switch is electrically connected to the "Interlock In/Out" port on the LIBSCAN spectrometer console via the 9-pin connector located directly above the laser aperture. The "Interlock In/Out" port connects with the laser power supply via the "Interlock Override Unit" using the supplied lead. Note that the "Interlock Override Unit" is specific to the laser being used – contact Applied Photonics Ltd for further information on this. Removal of the sample chamber from the LIBSCAN head, or disconnection of the Interlock In/Out lead, will also activate the interlock (ie. prevent activation of the laser). The key-switch operated Interlock Override Unit (see section 3.6 of this User's Manual) allows the safety interlock feature to be disabled. It is necessary to use this override feature when operating the LIBS module without a sample chamber (ie. operating in "open beam" mode). In view of this, the LIBSCAN system **must be categorised as a Class 4 laser product** since, by design, the product may be used in such a way that the laser beam is not contained (ie. "open beam" mode of operation)**.** If, however, the sample chamber is correctly fitted to the LIBS module and the Interlock Override Unit is switched off and the key removed, then the laser radiation is adequately contained to Class 1 Accessible Emission Limits by the design of the hardware.

IMPORTANT

- READ and UNDERSTAND both this User's Manual and the instructions provided by the manufacturer of the laser before operating the LIBSCAN system.
- NEVER allow unauthorised and/or untrained persons operate the LIBSCAN system.
- Only suitably qualified and authorised persons should activate the Interlock Override Unit (IOU). The key should be removed from the IOU and held by the Laser Safety Officer when this feature is not required.
- ALWAYS use appropriate laser safety protective eyewear when operating the LIBSCAN system in "open-beam" configuration – you should seek advice from your Laser Safety Officer on this matter.
- ALWAYS switch the laser off when not in use and remove the key from the keyswitch of the laser power supply to prevent unauthorised activation.
- NEVER operate the LIBSCAN system in areas where explosive gas mixtures may be present.
- NEVER operate the LIBSCAN system with any access cover removed.
- NEVER place inside the sample chamber flammable liquids or any other material which may give rise to flammable / explosive gas mixtures. Activation of the laser under these conditions could

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result in an explosion leading to severe personal injury and/or fire hazard. Remember that the laser-induced plasma is a source of ignition.

- NOTE that neither the opaque black Delrin nozzle aperture nor the transparent acrylic nozzle aperture provide any protection to the user against exposure to direct or scattered laser radiation. The function of the nozzle aperture is described later in this User's Manual.
- ALWAYS thoroughly inspect the LIBSCAN system for damage prior to use. Particular attention should be given to the laser safety windows and the electrical safety interlock fitted to the door of the sample chamber.
- NEVER point the LIBSCAN head at a person (even with laser switched off), especially towards the eyes, even if the person is wearing laser safety eyewear. **The laser should be considered "active" unless the laser power supply is deactivated and the safety shutter located on the side of the LIBSCAN head is switched to the CLOSED position**.

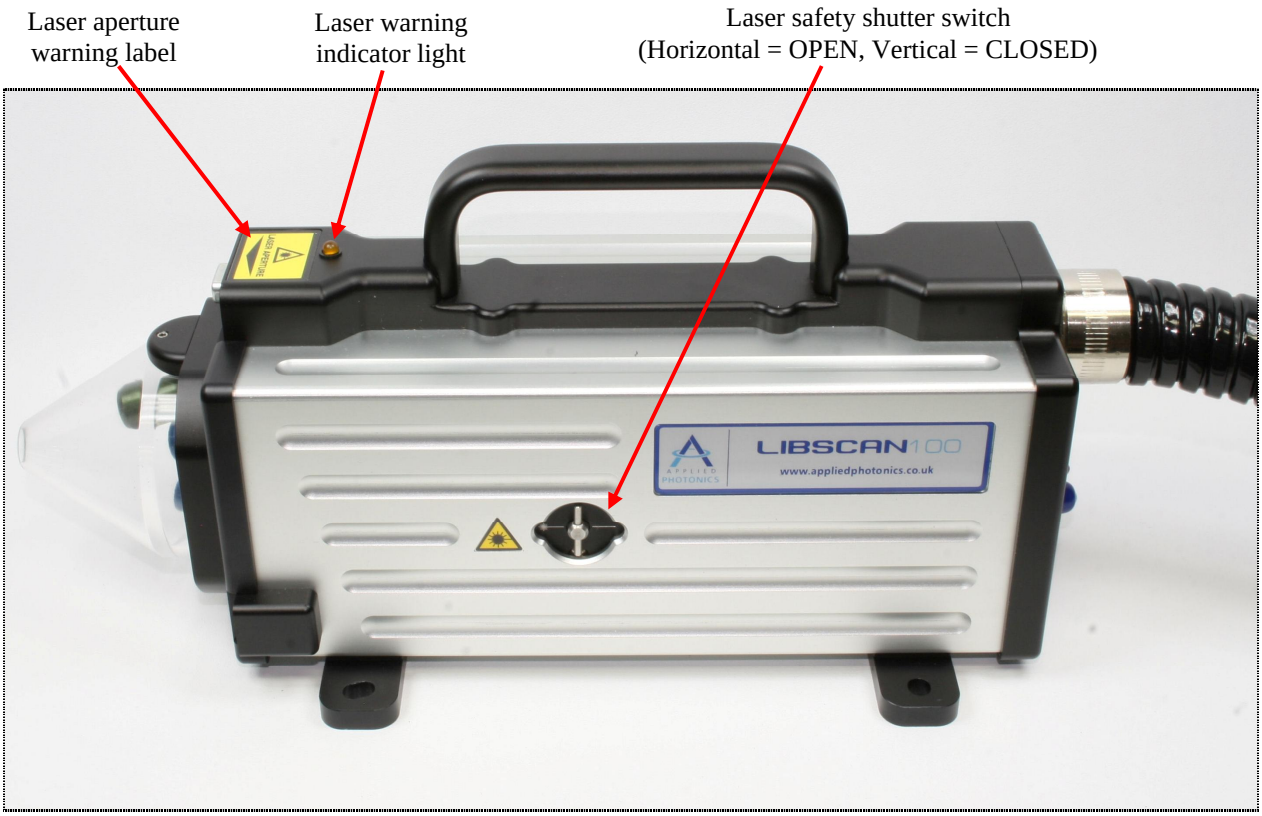
Laser aperture warning label
Laser warning indicator light
Laser safety shutter switch (Horizontal = OPEN, Vertical = CLOSED)
LIBSCAN100
www.appliedphotonics.co.uk

Image of LIBSCAN head showing location of laser shutter switch, warning indicator light and laser aperture warning label

2.2 Note on the laser safety window material used in the modular sample chambers

The laser safety window material fitted to the modular sample chambers is rated as follows:

Laser Wavelength (nm)	USA Standard ANSI Z 136.1 – 2000	UK & European Standard BS EN 207:1999
	Optical Density	Protection Level R (Q-switched laser)
1064	OD 6	L6
355	OD 5	L5
266	OD 3	L3

R L6 at 1064 nm indicates a protection level of maximum spectral transmittance of 10^{-6} at 1064 nm for a pulsed laser of pulse length 10^{-9} – 10^{-7} seconds (ie. a Q-switched laser).

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The laser safety windows used in Applied Photonics Ltd's range of modular sample chambers provide adequate protection against scattered laser light of wavelength 1064 nm, 355 nm and 266 nm such that Class 1 Accessible Emission Limits are met if the sample chamber is used correctly and laser pulse energies are not excessively high (ie. typically less than 250 mJ with a 5 - 10 ns pulse length). Given that the modular sample chambers may be used with a variety of LIBSCAN system (and LIBS-6 and LIBS-8 integrated LIBS modules) configurations, it is the responsibility of the user to conduct a risk assessment to establish whether or not the laser safety windows provide adequate protection for the particular laser being used. If in any doubt, you should consult your Laser Safety Officer and/or Applied Photonics Ltd for advice on this matter.

Warning – the Applied Photonics Ltd modular sample chambers are designed specifically for use with our LIBSCAN (and our LIBS-6 / LIBS-8 integrated LIBS modules) range of products and should not be used with any other laser device or product. If in doubt, seek advice from the manufacturer, Applied Photonics Ltd.

2.3 Electrical

The LIBSCAN system contains electrical circuits operating at 12 VDC at a maximum current of 2.5 Amp. Accordingly, they pose no electric shock risk. The laser head (located inside the LIBSCAN head) and associated power supply, however, contain electrical circuits operating at potentially lethal voltage and current levels. Consult the manufacturer's User Manual supplied with the laser for further guidance on the safe use of the laser.

3 General description

3.1 Overview

The main components of LIBSCAN 50 / LIBSCAN 100 (with SC-2C sample chamber fitted) are illustrated in the following figure.

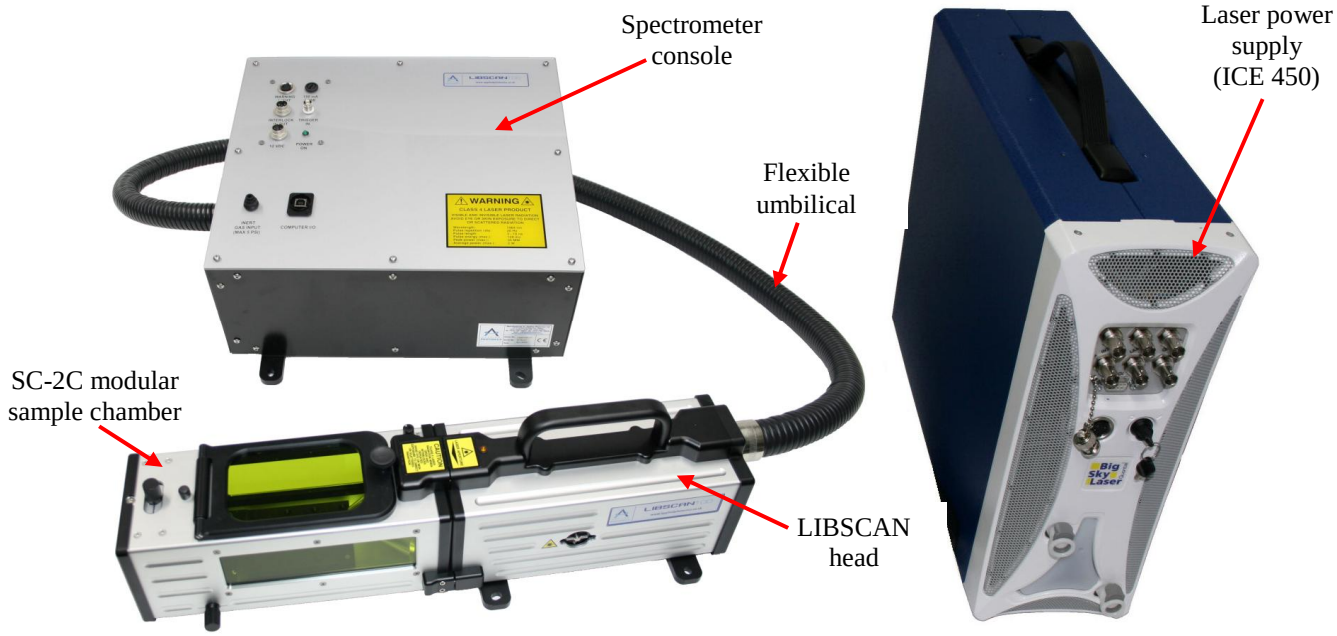

Image showing spectrometer console, LIBSCAN head, SC-2C sample chamber and ICE 450 laser power supply

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3.2 Spectrometer console

The spectrometer console contains the optical spectrometers (up to six or eight spectrometers may be installed, depending on the model of LIBSCAN system) and provides the interface between the LIBSCAN head and i) the laser power supply and ii) the personal computer. The console requires a 12 Volt DC electrical supply (2.5 Amps max.) which is derived from the mains electrical supply using the low-voltage adaptor (12 VDC, 2.5 Amps) supplied with LIBSCAN.

External warning light (Output = 15 VDC, 150 mA max.). Fuse located on right
Interlock IN / OUT (connects to "Interlock Override Unit" fitted to the top of the ICE 450 laser power supply)
12 Volt DC power input (<2.5 Amp)
Inert gas inlet port (requires 4 mm OD plastic tubing) for gas-purge of sample
Spectrometer trigger input (connects to "Q-Switch Out" BNC port on laser power supply)
"Power On" indicator light
USB 2.0 connection for external computer
LIBSCAN100
www.appliedphotonics.co.uk
WARNING LIGHT
150 mA FUSE
INTERLOCK IN/OUT
TRIGGER IN
12 VDC
POWER ON
INERT GAS INPUT (MAX 5 PSI)
COMPUTER I/O
WARNING
CLASS 4 LASER PRODUCT
VISIBLE AND INVISIBLE LASER RADIATION
AVOID EYE OR SKIN EXPOSURE TO DIRECT OR SCATTERED RADIATION

Close-up view of the spectrometer console

3.3 LIBSCAN head

The LIBSCAN head contains the laser and associated optics required to focus the laser beam on to a sample and collect the plasma light for transmission to the spectrometers located within the spectrometer console. Two versions are available – 6-channel and 8-channel. The 6-channel version is illustrated throughout this User's Manual. The head is designed to accommodate a miniature CCD camera and associated components used with the optional imaging kit. The main features of the LIBSCAN head fitted with the optional imaging kit are illustrated in the following figures. Use of the transparent aperture nozzle allows the operator to view the laser plasma even when a sample is placed up against the aperture. **WARNING: Note that neither the transparent nor the opaque aperture nozzle provides any protection to the user against exposure to the laser radiation.**

It is not necessary to have an aperture nozzle fitted to the LIBSCAN head although it is beneficial to do so as it provides the following functions: i) facilitates correct positioning of the sample being analysed, ii) helps to direct the purge gas (if used) to the region in space where the laser plasma will be formed, and iii) provides protection for the optics etc against accidental mechanical damage.

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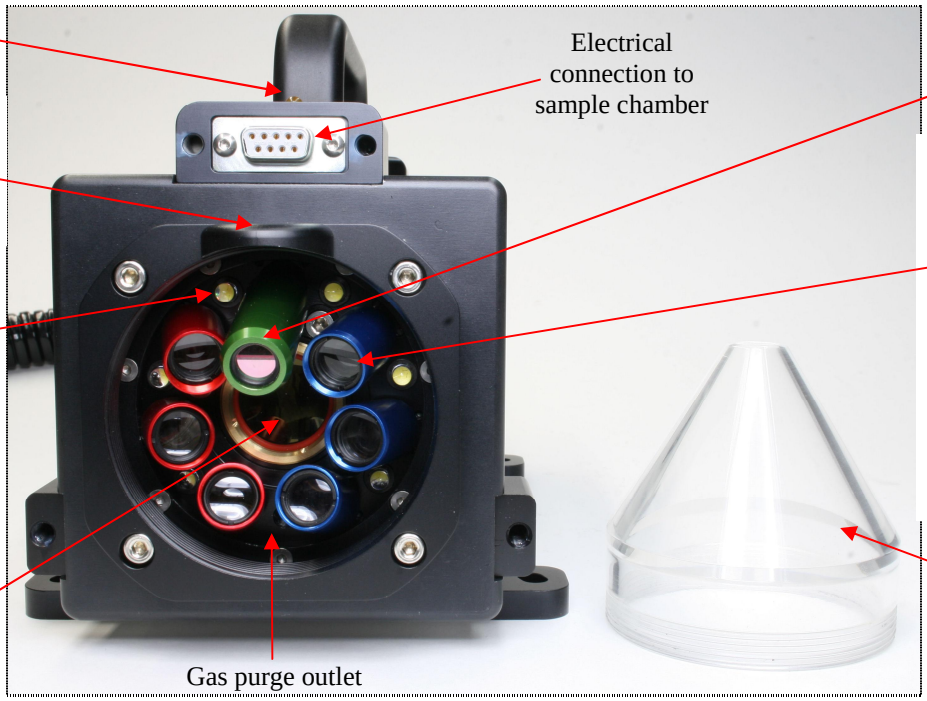
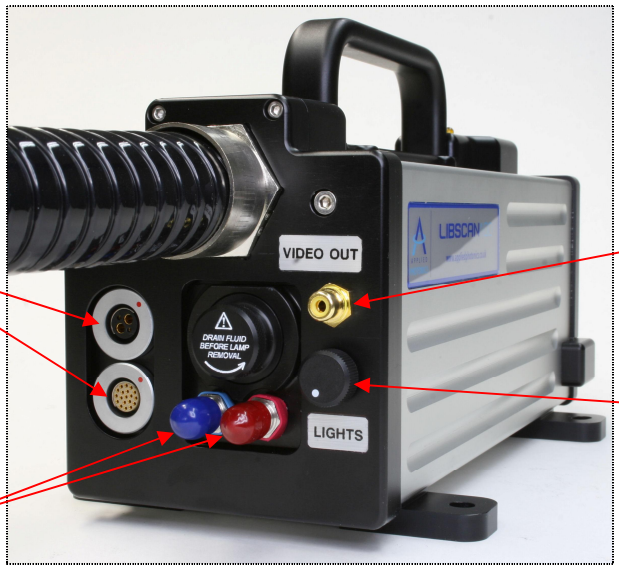
LIBSCAN head with aperture nozzle removed

LIBSCAN head with transparent aperture nozzle fitted


Laser warning indicator light
Grub screw for locking aperture nozzle (do not over-tighten)
Circular array of high-brightness white LED lights (optional feature)
Laser beam expander and output aperture
Gas purge outlet
Electrical connection to sample chamber
Miniature CCD camera (optional feature)
Circular array of six lens holders – used to collect plasma light. The blue lens holders are for collection of UV-VIS while the red lens holders are for collection of VIS-NIR. The 8-channel version has 4 UV-VIS (blue) and 4 VIS-NIR (red) lens holders.
Transparent aperture nozzle

Front view of 6-channel LIBSCAN head showing plasma light collection lens array, CCD camera, LED lights, laser aperture and gas purge outlet



Rear view of LIBSCAN head showing electrical / water connections to laser head and electrical connection for the camera

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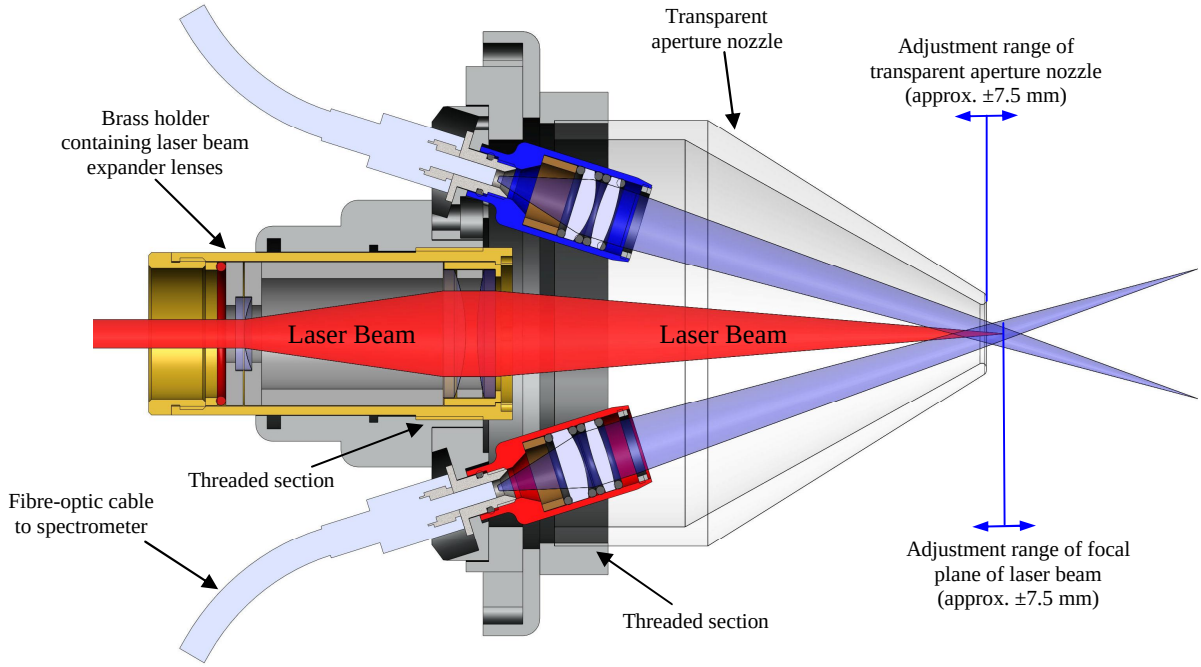
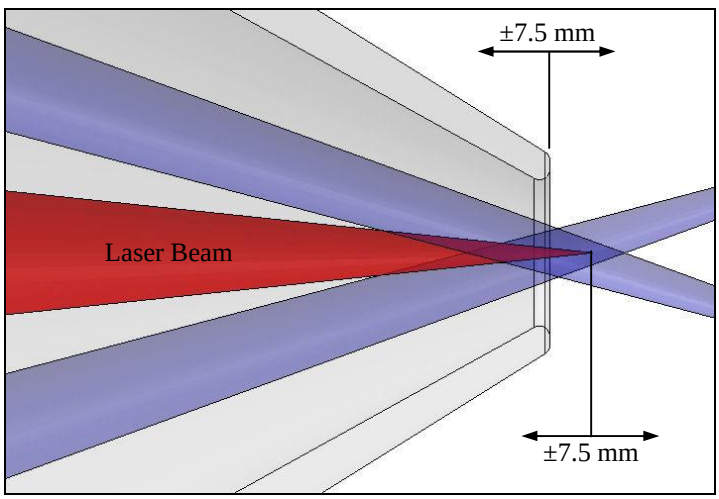
3.4 Laser beam expander and plasma light collection optics

The optical configuration used in the LIBSCAN system is illustrated schematically in the following diagram. The laser beam expander consists of three lenses and is used to provide a tightly focussed laser beam at nominally 80 mm from the aperture of the beam expander (90 mm for the 8-channel LIBSCAN). The brass tubular piece which houses the beam expander lenses is threaded so that rotating it causes the focal plane of the laser beam to move. A special tool is used to rotate the brass tubular piece after first removing the nozzle aperture for access. The design allows for approximately ±7.5 mm of adjustment, as illustrated in the following diagrams. The plasma light collection optics are angled at approx. 17.4 degrees (15.7 degrees for the 8-channel LIBSCAN) and are designed to collect light from the region in space defined in the following diagram. The transparent aperture nozzle is threaded so that rotation causes it to move along the optic axis of the laser beam – range of travel is approximately ±7.5 mm (it may be removed from the body of the LIBS module by unscrewing further). The main purpose of the transparent aperture nozzle is to provide a convenient means of setting the distance to the sample surface. The nozzle aperture also provides containment of the purge gas to ensure the sample surface and laser-induced plasma region are effectively purged of atmospheric air, and provides physical protection for the laser and plasma light collection optics (and imaging camera if fitted).


Transparent aperture nozzle
Adjustment range of transparent aperture nozzle (approx. ±7.5 mm)
Brass holder containing laser beam expander lenses
Laser Beam
Laser Beam
Threaded section
Fibre-optic cable to spectrometer
Adjustment range of focal plane of laser beam (approx. ±7.5 mm)
Threaded section



Close-up view of laser beam focus and field-of-view of plasma light collection optics

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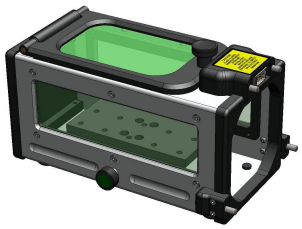
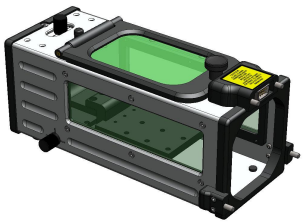
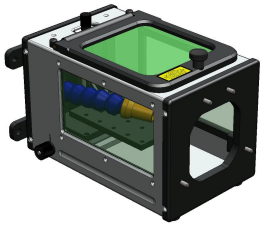
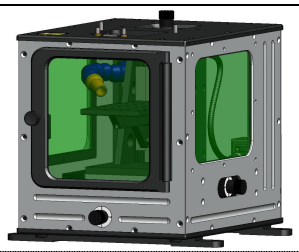
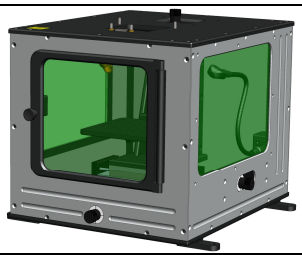
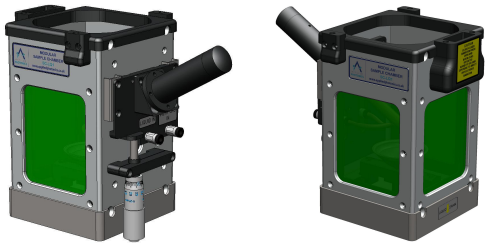
3.5 Modular sample chambers

A range of modular sample chambers is available for use with the LIBSCAN system (see table below). These sample chambers are also suitable for use with our LIBS-6 and LIBS-9 integrated LIBS modules. Two types (SC-2C and SC-2L) are described in more detail in the remainder of this User's Manual.

			
SC-1	SC-2C	SC-2M	SC-2L
Single axis translation stage, 20 mm travel per stage (manual control). Approx. overall dimensions: 110 x 120 x 200 mm	2-axis translation stage, 20 mm travel per stage (manual control). Approx. overall dimensions: 110 x 120 x 250 mm	2-axis translation stage, 20 mm travel per stage (manual control) Fume extract port. Internal LED light. Approx. overall dimensions: 170 x 170 x 270 mm	3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 260 x 260 x 320 mm
			
SC-2XL	SC-3L	SC-LQ1	
3-axis translation stage, 50 mm travel per stage (manual control). Fume extract port. Internal LED light. Approx. overall dimensions: 475 x 370 x 450 mm	3-axis translation stage, 50 mm travel per stage (XY computer-controlled). Fume extract port. Internal LED light. Approx. overall dimensions: To Be Advised.	Modular sample chamber designed to analyse liquids. Requires inert gas and flow of liquid to be connected to chamber via two ports designed to accept 4 mm flexible tubing. Liquid drain port in base of chamber. Micrometer control of position of liquid surface relative to focal plane of laser beam. Approx. overall dimensions: 225 x 245 x 200 mm	

Current range of modular sample chambers manufactured by Applied Photonics Ltd

WARNING

The modular sample chambers described above are designed specifically for use with our LIBSCAN range of modular LIBS systems and our LIBS-6 / LIBS-8 integrated LIBS modules. The specifications for the laser safety windows are as follows:

Laser Wavelength (nm)	**USA Standard ANSI Z 136.1 – 2000**	**UK & European Standard BS EN 207:1999**
	Optical Density	**Protection Level R (Q-switched laser)**
1064	OD 6	L6
355	OD 5	L5
266	OD 3	L3

R L6 at 1064 nm indicates a protection level of maximum spectral transmittance of 10^{-6} at 1064 nm for a pulsed laser of pulse length 10^{-9} – 10^{-7} seconds (ie. a Q-switched laser).

Since the LIBS-6 / LIBS-8 integrated LIBS modules may be used with various laser devices, it is the responsibility of the user to establish whether the protection offered by the laser safety windows is adequate for the laser being used. If in doubt, seek advice from a suitably qualified Laser Safety Officer or contact Applied Photonics Ltd before operating the laser equipment.

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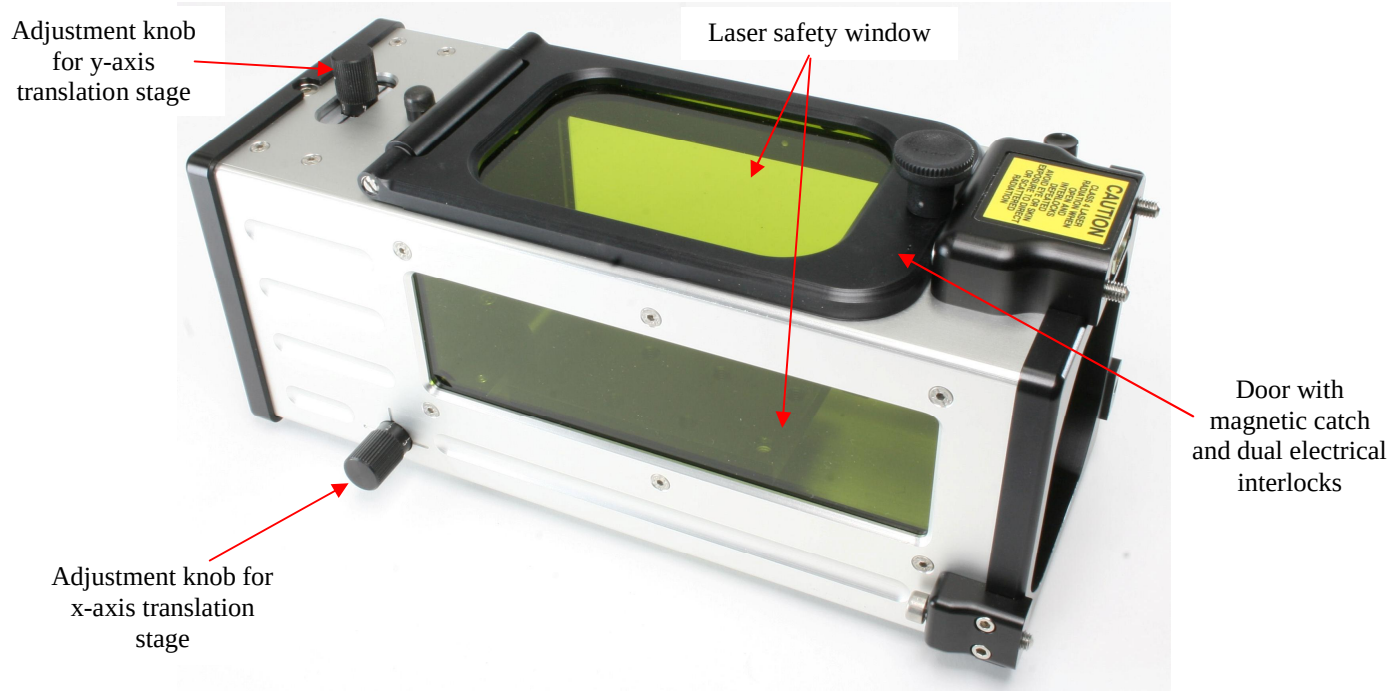
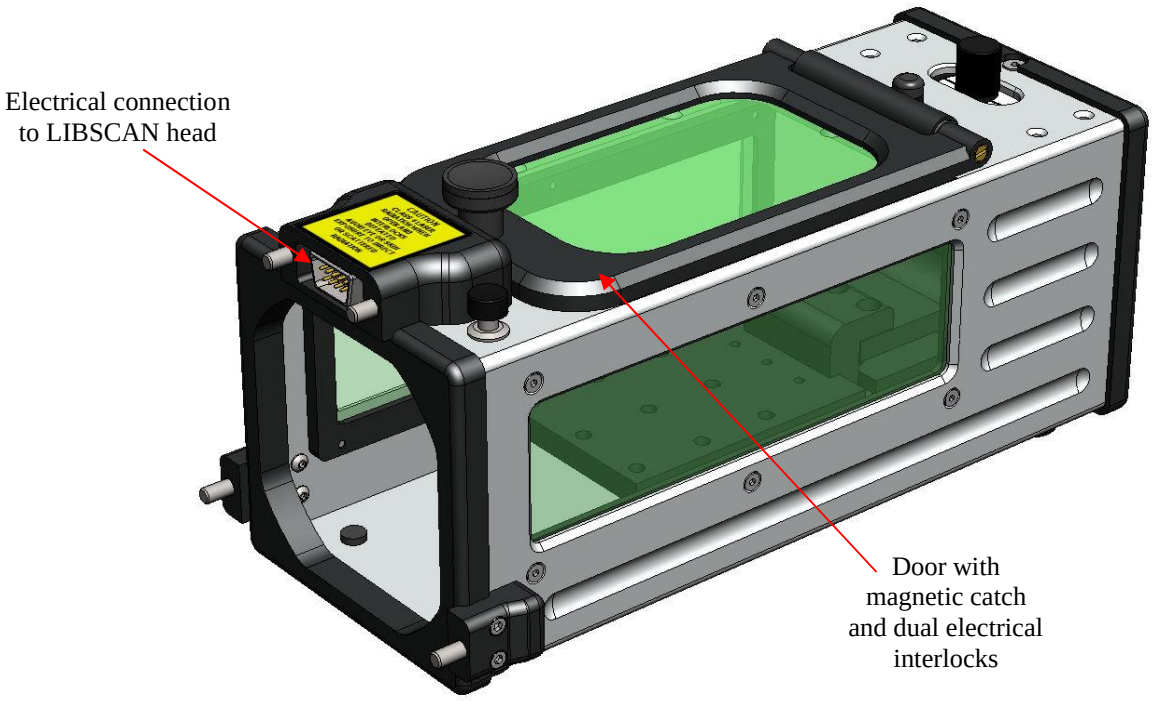
3.5.1 SC-2C modular sample chamber

A general view of the SC-2C modular sample chamber is given in the following figure. The sample chamber is equipped with a breadboard plate which is attached to a manual two-axis translation stage (approx. 20 mm travel per stage). The breadboard plate has an array of M6 tapped (blind) holes on 25 mm centres and which may be used to facilitate the attachment of a sample holder etc. Movement of the breadboard is achieved by adjustment of the knobs on the side and top of the sample chamber, as illustrated below.


Adjustment knob for y-axis translation stage
Laser safety window
Door with magnetic catch and dual electrical interlocks
Adjustment knob for x-axis translation stage
CAUTION

View of SC-2C modular sample chamber

The sample chamber is equipped with a number of features as illustrated in the following figures.



CAD view of SC-2C modular sample chamber

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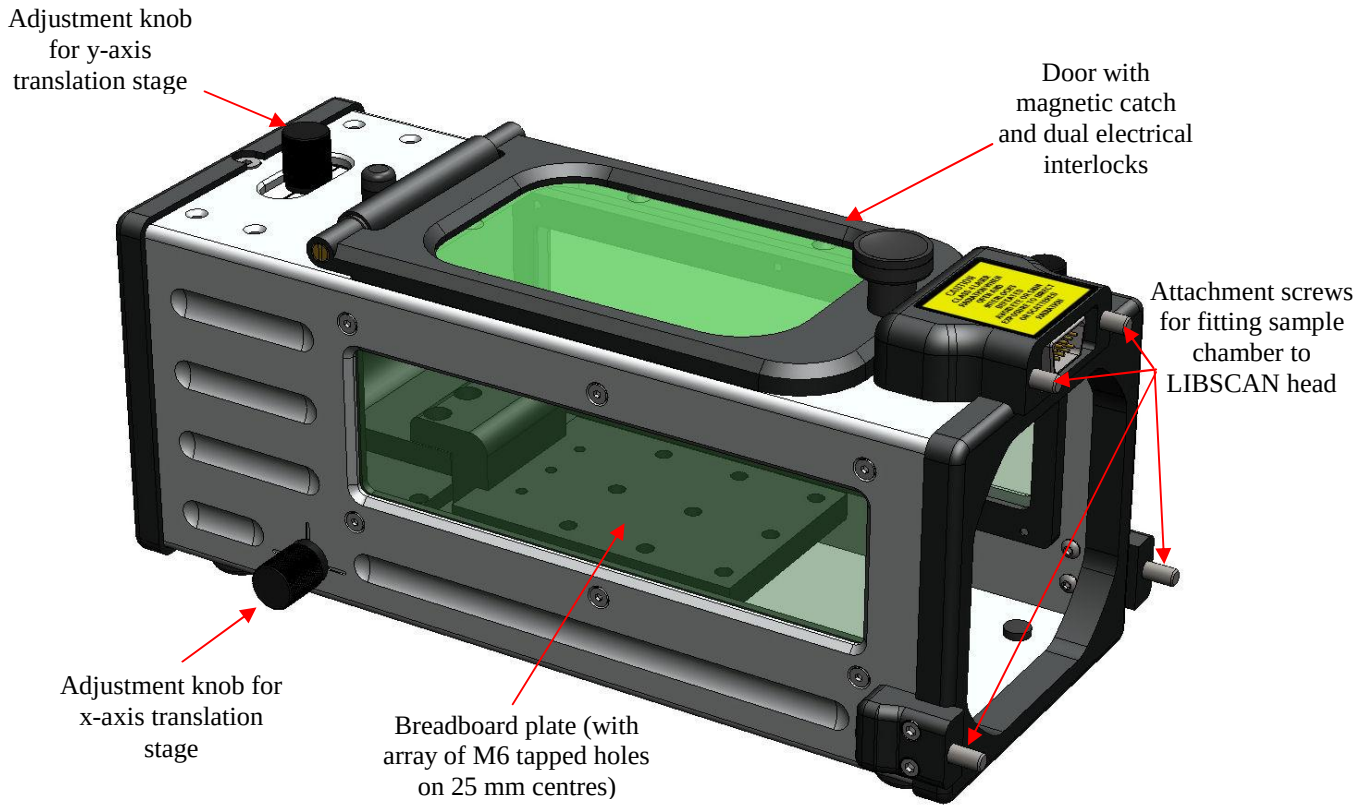
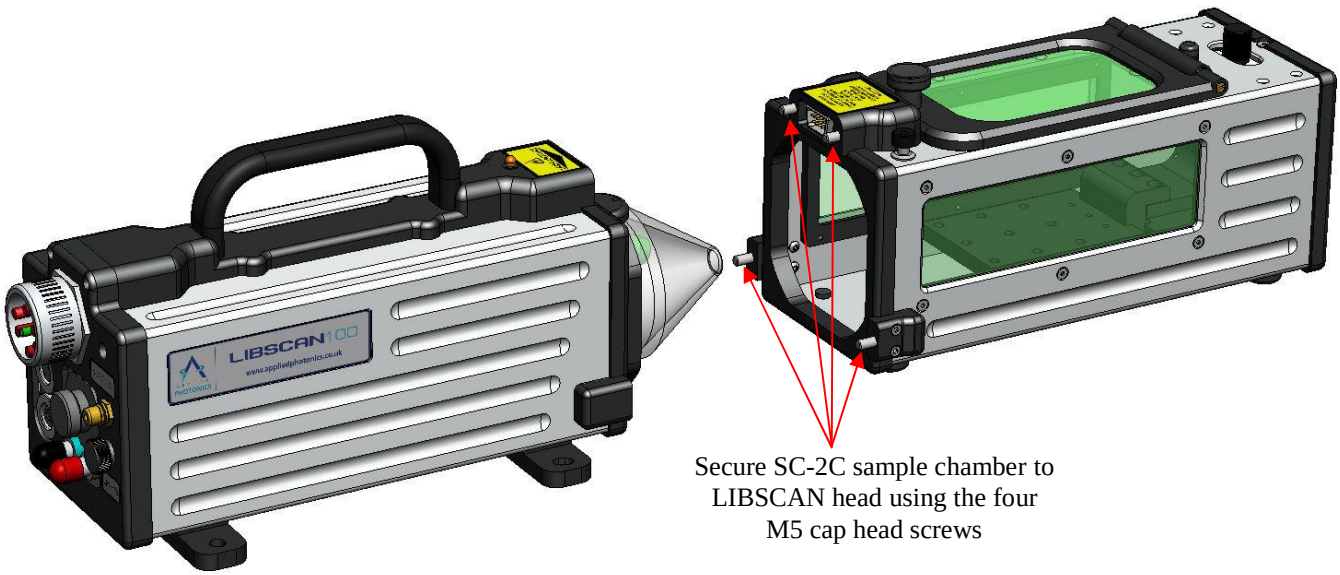

Adjustment knob for y-axis translation stage
Door with magnetic catch and dual electrical interlocks
Attachment screws for fitting sample chamber to LIBSCAN head
Adjustment knob for x-axis translation stage
Breadboard plate (with array of M6 tapped holes on 25 mm centres)

CAD view of SC-2C modular sample chamber

The SC-2C modular sample chamber is designed to fit to the LIBSCAN head using four M5 screws as illustrated in the following figures. Before fitting the sample chamber to the LIBSCAN head, ensure that the breadboard plate is positioned sufficiently low so as not to make contact with the aperture nozzle of the LIBSCAN head (use the y-axis adjustment knob to lower the breadboard plate). Be careful to fit the sample chamber squarely to the LIBSCAN head so as not to cause damage to the electrical connectors.



CAD views of SC-2C modular sample chamber illustrating method of attachment to LIBSCAN 100 head

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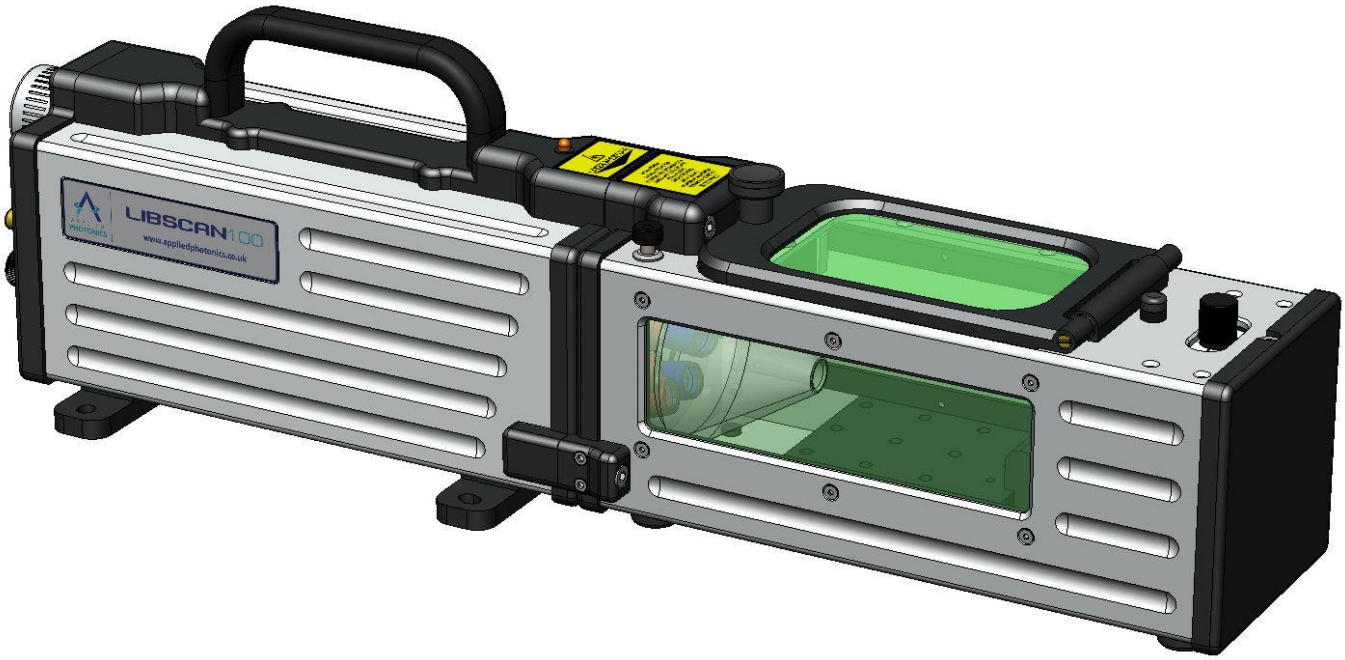

CAD views of SC-2C modular sample chamber attached to LIBSCAN 100 head

3.5.2 SC-2L modular sample chamber

A general view of the SC-2L modular sample chamber is given in the following figure. The sample chamber is equipped with a breadboard plate which is attached to a manual three-axis translation stage (50 mm travel per stage). The breadboard plate has an array of M6 tapped (blind) holes on 25 mm centres and which may be used to facilitate the attachment of a sample holder etc. Movement of the breadboard is achieved by adjustment of the knobs on the sides and top of the sample chamber, as illustrated below.

Adjustment knob for z-axis translation stage
Door with magnetic catch and dual electrical interlocks
Adjustment knob for x-axis translation stage
Laser safety window
Adjustment knob for y-axis translation stage

View of SC-2L modular sample chamber

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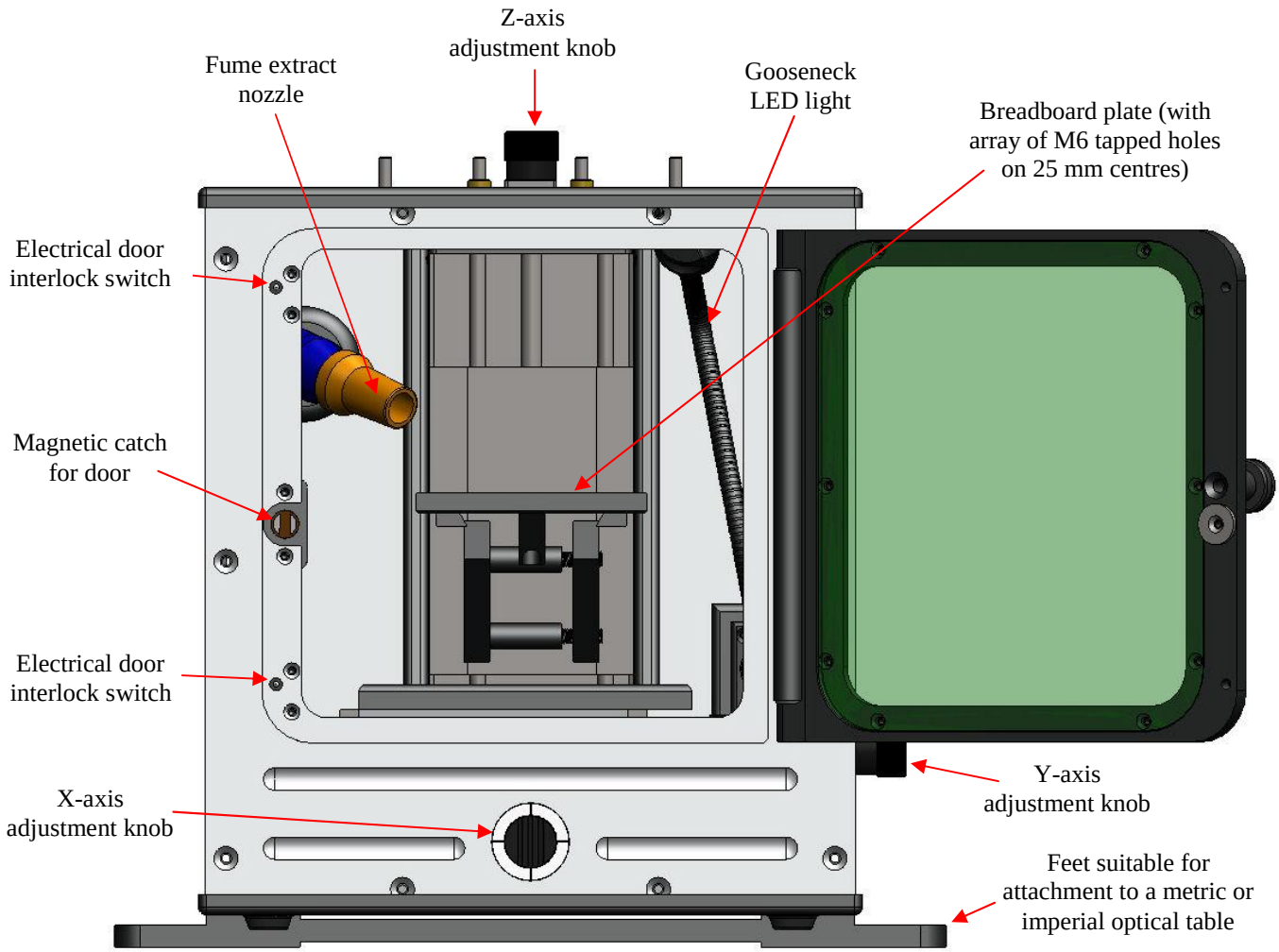
The sample chamber is equipped with a number of features as illustrated in the following figures.

CAD views of SC-2L modular sample chamber



CAD view of SC-2L modular sample chamber

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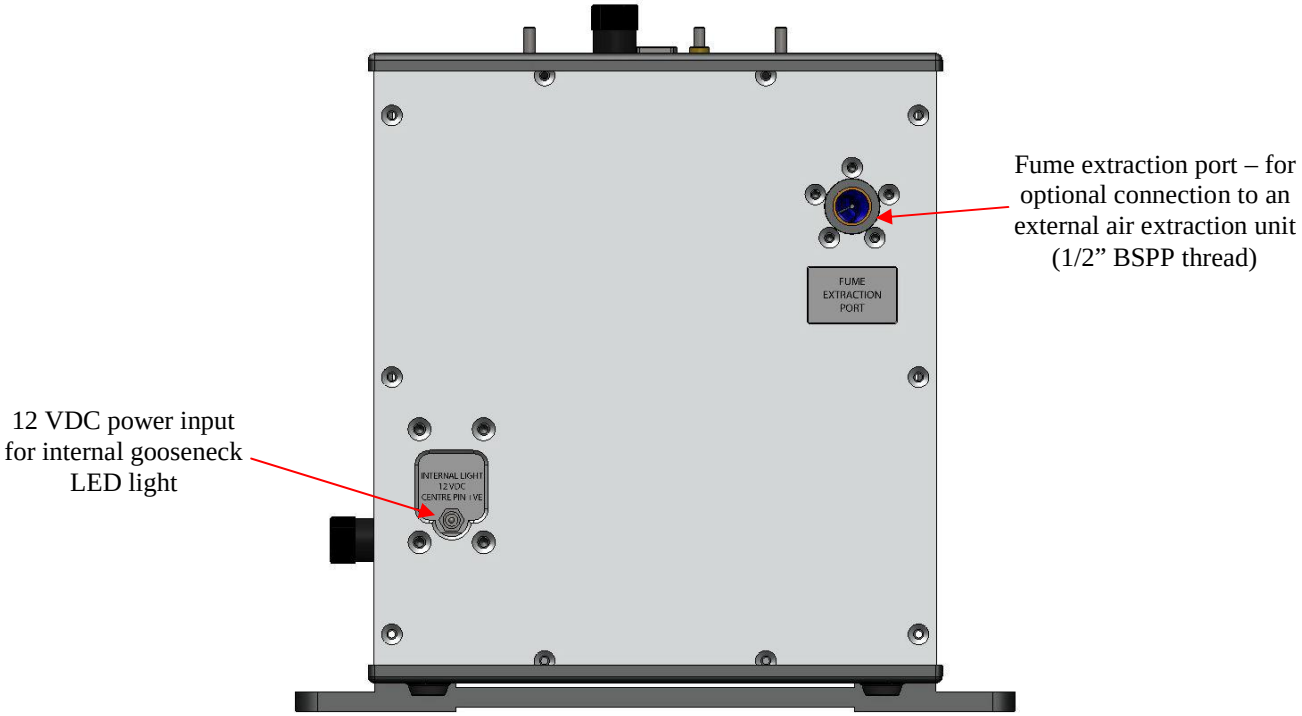
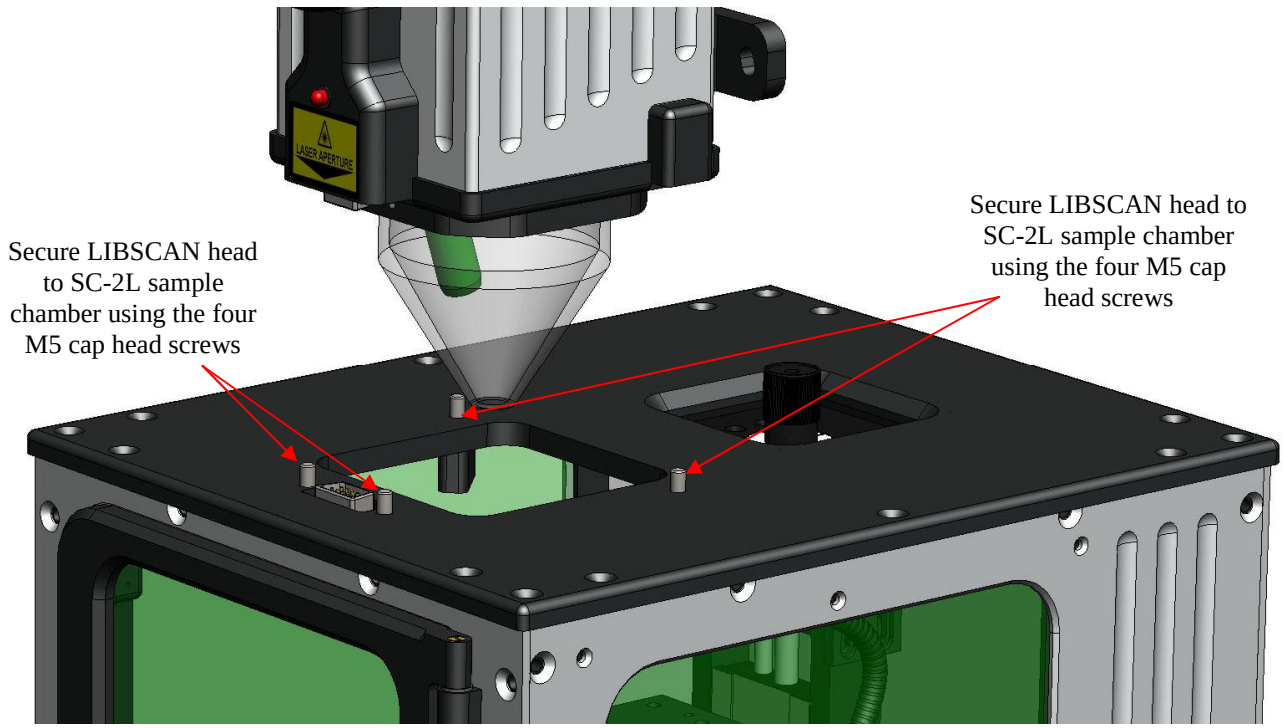

Fume extraction port – for optional connection to an external air extraction unit (1/2” BSPP thread)
FUME EXTRACTION PORT
12 VDC power input for internal gooseneck LED light
INTERNAL LIGHT 12 VDC CENTRE PIN +VE

CAD view of rear panel of SC-2L modular sample chamber

The LIBSCAN Head is designed to fit to the top of the sample chamber using four M5 screws as illustrated in the following figures. Before fitting the LIBSCAN head to the sample chamber, ensure that the breadboard plate is positioned sufficiently low so as not to make contact with the aperture nozzle of the LIBSCAN head. Be careful to fit the LIBSCAN head squarely to the sample chamber so as not to cause damage to the electrical connectors.


Secure LIBSCAN head to SC-2L sample chamber using the four M5 cap head screws
Secure LIBSCAN head to SC-2L sample chamber using the four M5 cap head screws
LASER APERTURE

CAD view of SC-2L modular sample chamber illustrating method of attachment of LIBSCAN head

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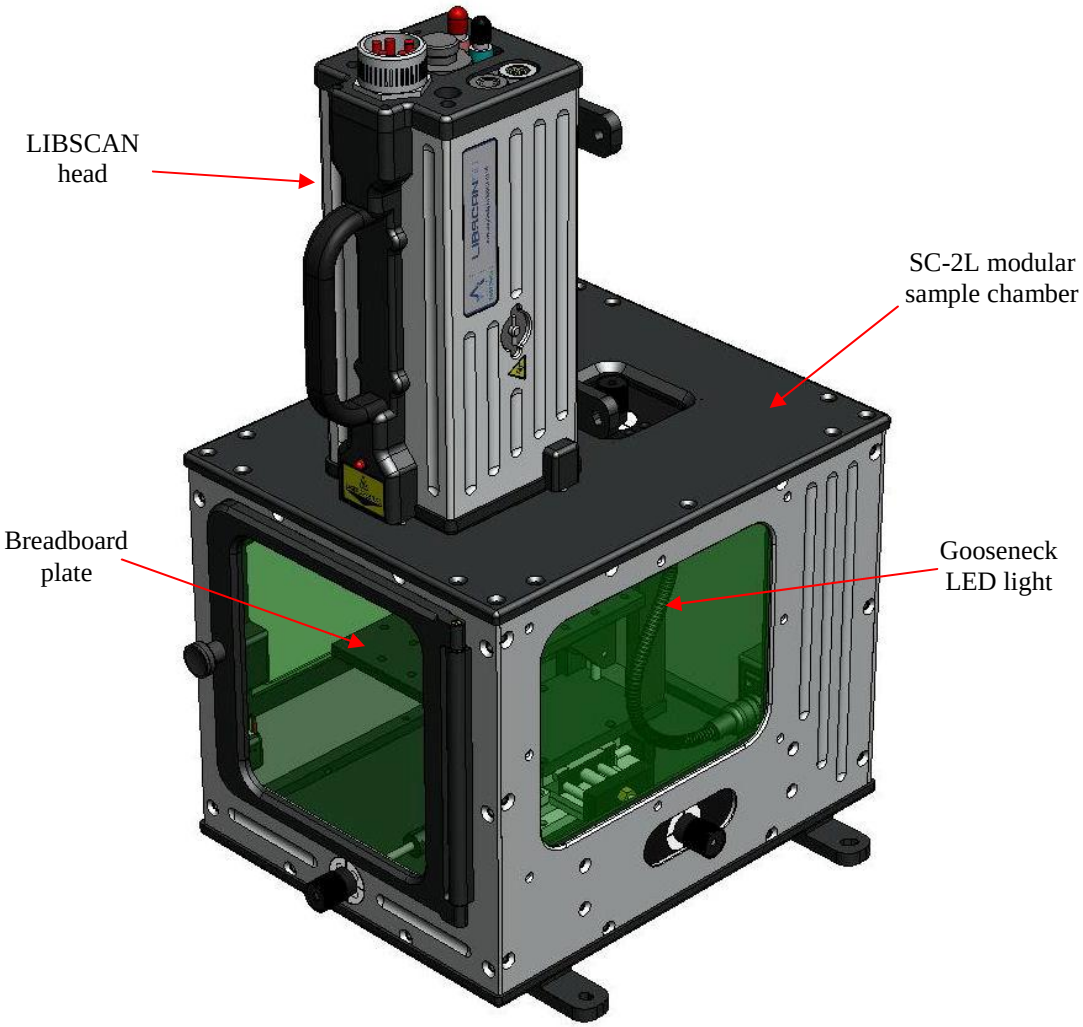
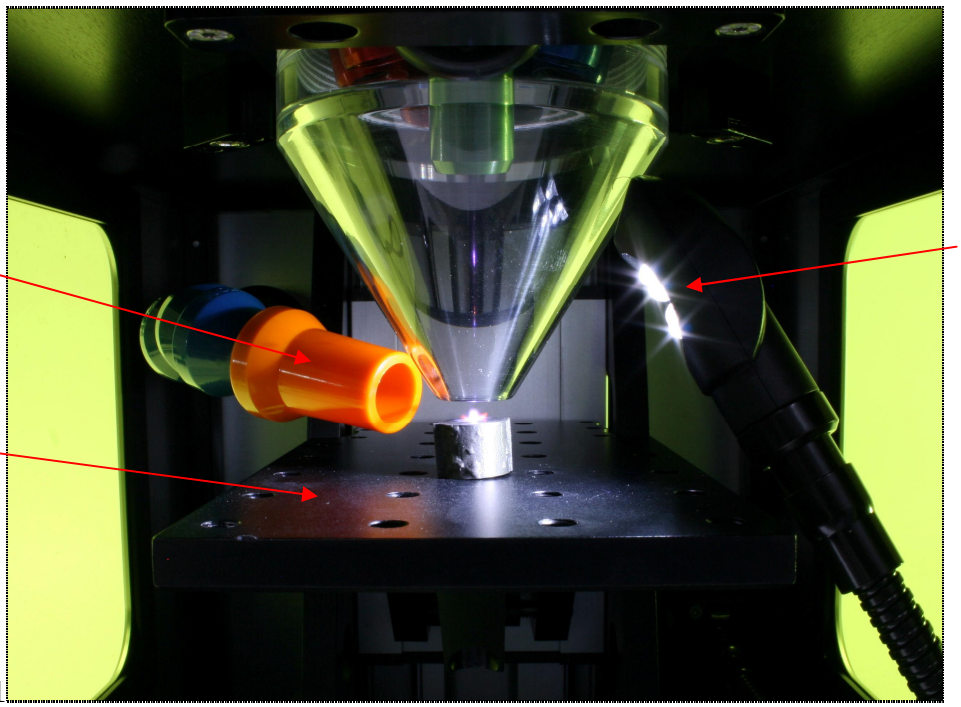


CAD view of LIBSCAN Head fitted to SC-2L modular sample chamber



Close-up view of inside of SC-2L sample chamber with LIBSCAN head fitted and showing a laser-induced plasma on a metallic target

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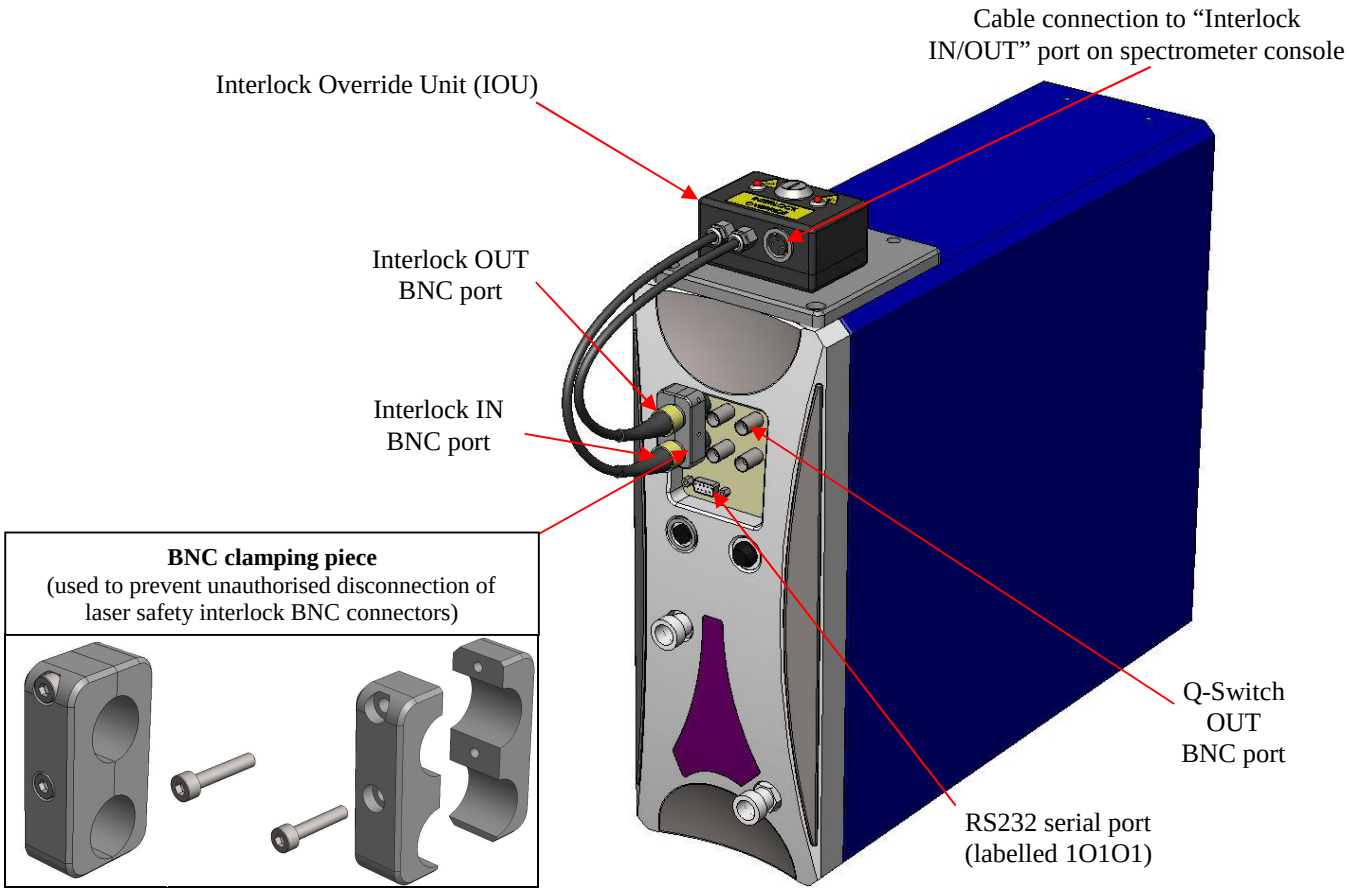
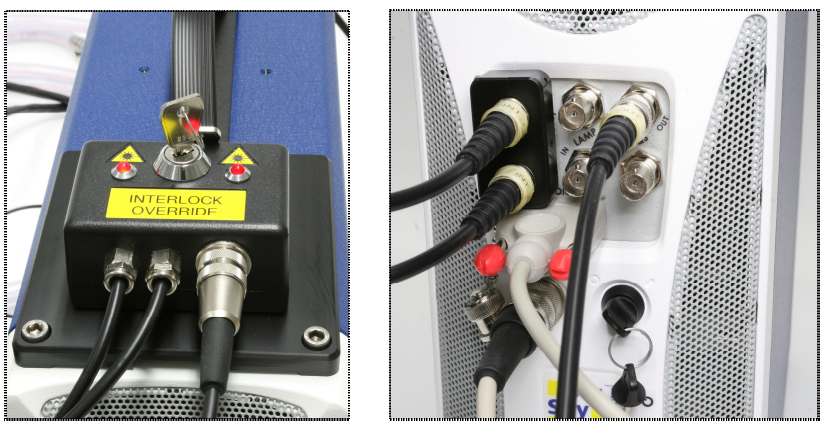
3.6 ICE 450 laser power supply and Interlock Override Unit

See Quantel's instructions for a complete description of the ICE 450 laser power supply and cooling group unit. On initial unpacking of the LIBSCAN system, the Interlock Override Unit (IOU) will need to be fitted to the ICE 450 laser power supply as illustrated in the following diagram.


Cable connection to "Interlock IN/OUT" port on spectrometer console
Interlock Override Unit (IOU)
Interlock OUT BNC port
Interlock IN BNC port
BNC clamping piece
(used to prevent unauthorised disconnection of laser safety interlock BNC connectors)
Q-Switch OUT BNC port
RS232 serial port (labelled 1O1O1)

Schematic diagram illustrating Interlock Override Unit and electrical connections to ICE 450 laser power supply

The Interlock Override Unit is used to override the laser safety interlock when the LIBSCAN system is required to be used in "open beam" configuration (ie. without a sample chamber). To override the interlock, the keyswitch should be turned "on" by turning clock-wise as indicated in the image below. The two red LEDs will flash continuously when the keyswitch is activated and the laser is in "active" mode. When the keyswitch is set to "off", the red LEDs will be extinguished indicating that the laser safety interlock is operating. The key should be removed from the Interlock Override Unit to prevent unauthorised activation of the interlock override.



Images illustrating Interlock Override Unit and electrical connections to ICE 450 laser power supply

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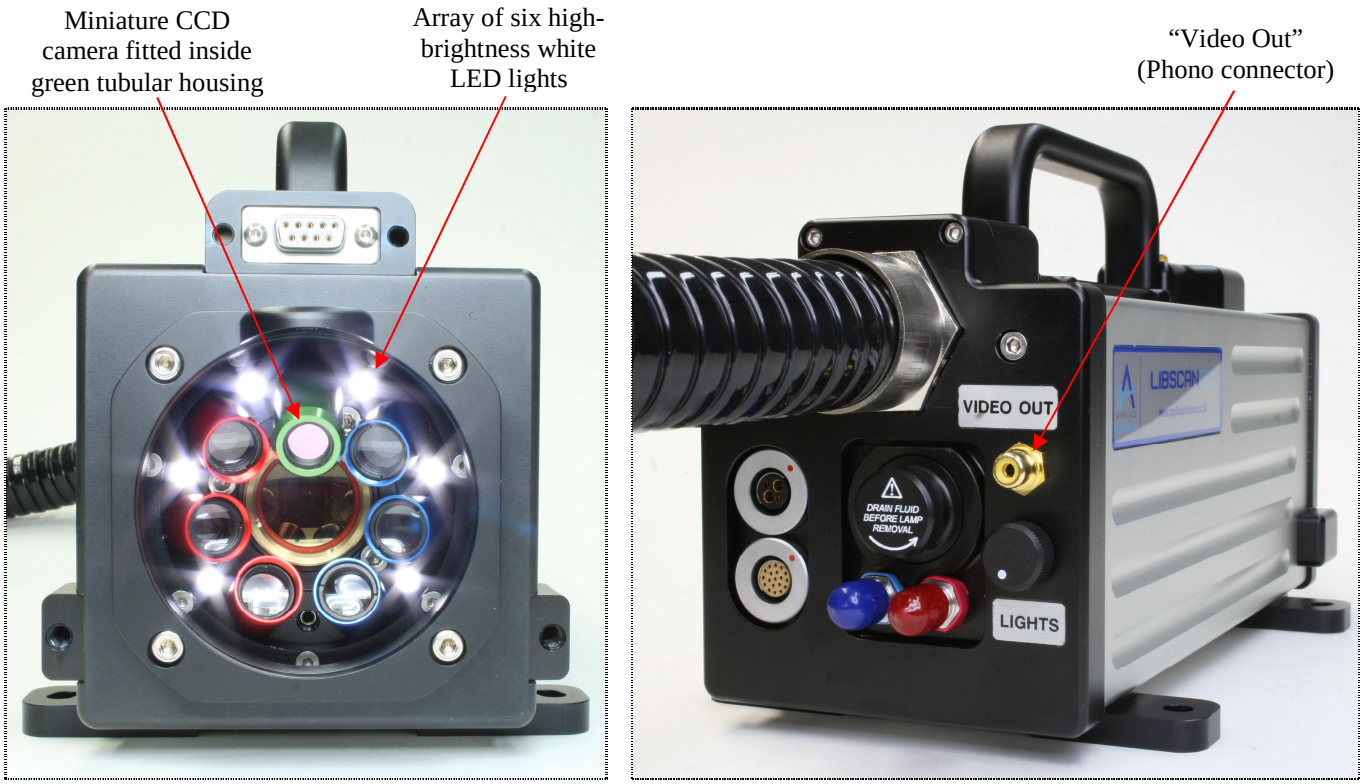
3.7 Imaging camera and associated components

The imaging camera is offered as an optional feature for LIBSCAN. A miniature colour CCD camera is installed inside the LIBSCAN head and is located within the green tubular holder at the 12 o'clock position at the front of the head (illustrated below). The tubular holder is fitted with an optical filter to protect the CCD camera from damage due to possible high levels of stray laser light (1064 nm). The electrical connections to the camera are accessible at the rear of the LIBSCAN head as illustrated below.


Miniature CCD camera fitted inside green tubular housing
Array of six high-brightness white LED lights
"Video Out" (Phono connector)
VIDEO OUT
LIBSCAN
DRAIN FLUID BEFORE LAMP REMOVAL
LIGHTS

Images of LIBSCAN head showing location of CCD camera and lights (front) and "Video Out" phono connector (rear)

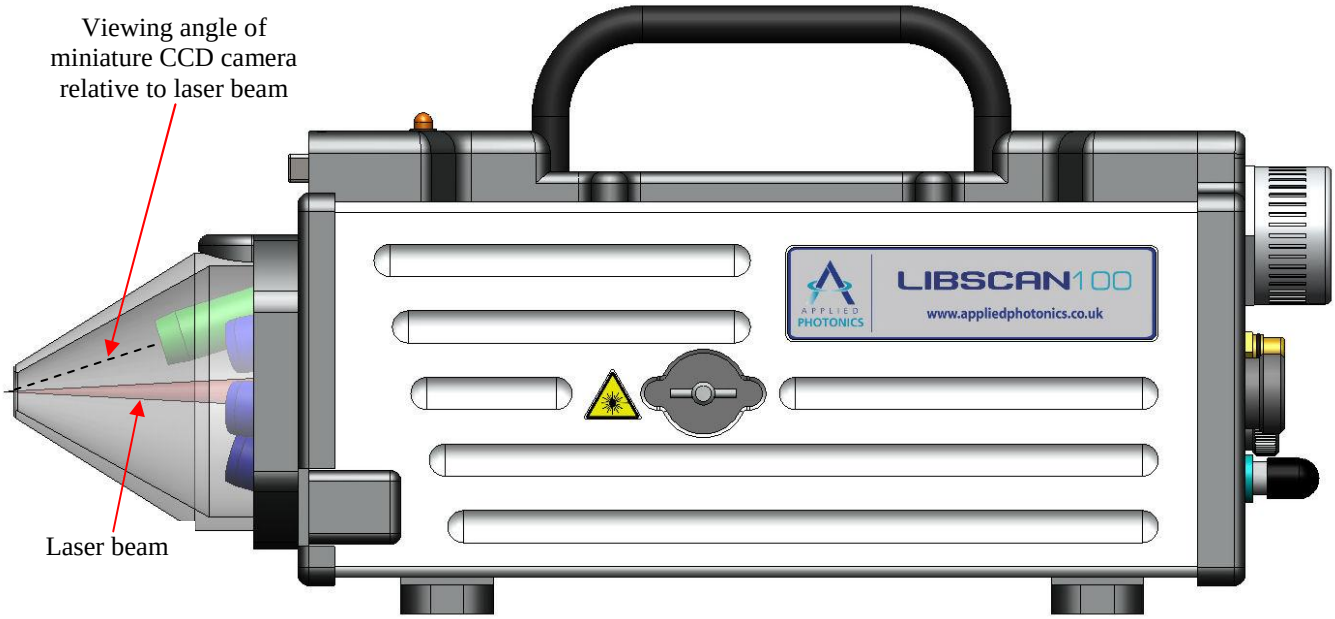


Image of LIBSCAN head showing viewing angle of miniature CCD camera

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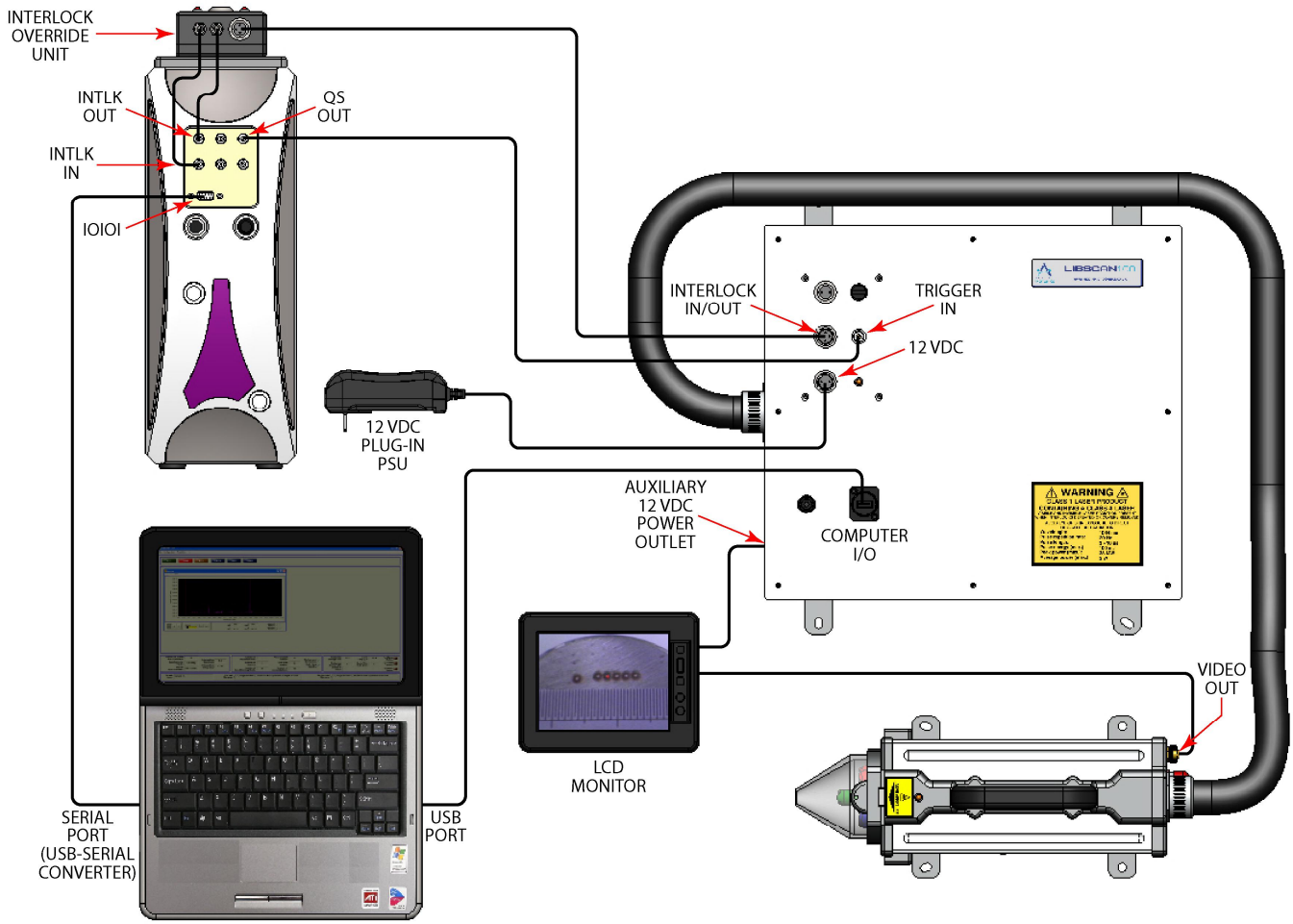
The specifications for the miniature CCD camera are:

- 1/7-inch CCD colour sensor (340,000 pixels)
- Field angle: approx. 51º horizontal, 42º vertical
- Micro f = 2.1 mm, f/2.9 lens
- Adjustable focus (approx. 20 mm to infinity)
- 12 VDC (110 mA)

An LCD colour monitor is supplied with the imaging camera kit and is connected as illustrated in the schematic diagram on the following section (section 3.8). Alternatively, the video images may be fed to a computer via the USB frame grabber also supplied with the imaging kit. A future version of APL's LIBSoft software will provide an imaging feature.

3.8 Low-voltage electrical connections

The following schematic diagrams illustrate the low-voltage (12 VDC power input), TTL trigger, external laser warning light (15 VDC output, 150 mA max.), laser safety interlock and computer USB connections between the various components of the LIBSCAN system. If the personal computer (PC) which is to be used with the LIBSCAN system is not equipped with an RS232 serial port, it will be necessary to use a Serial-to-USB converter lead to connect the ICE 450 laser power supply to the PC. A software driver is normally required which would be supplied with the Serial-to-USB converter lead. A suitable serial-to-USB converter lead is available from Applied Photonics Ltd as an optional item when ordering a LIBSCAN system.



Schematic diagram illustrating electrical connections between LIBSCAN components

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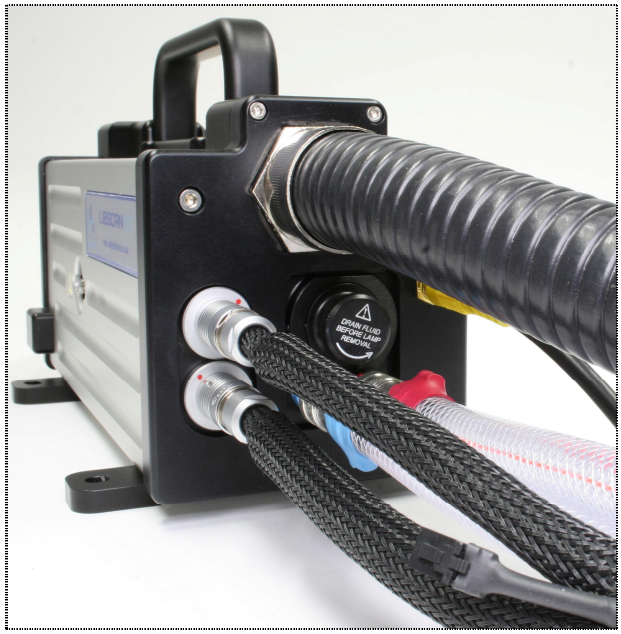
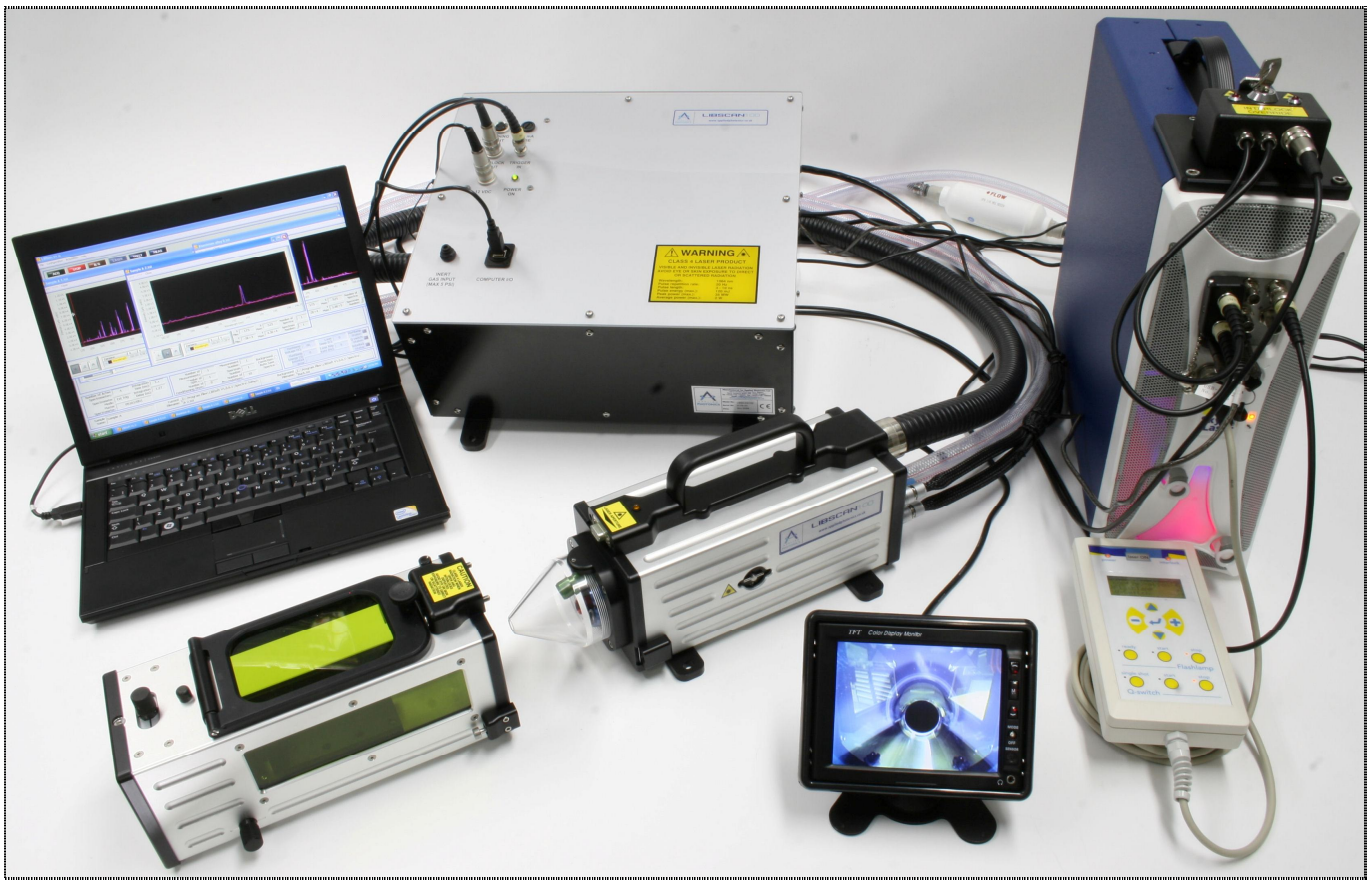
3.9 Assembly of LIBSCAN system

Follow Quantel's instructions for filling the ICE 450 laser power supply with cooling water and for connecting the water and electrical supplies to the laser head. Using the schematic diagram on the preceding page (section 3.8) make all electrical connections using the supplied leads (interconnecting leads supplied with the LIBSCAN system are labelled to facilitate final assembly). The following images illustrate how the LIBSCAN system should look after final assembly.





Images of LIBSCAN system after final assembly

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4 Operating procedure

Step 1 LIBSCAN should first be checked for obvious signs of damage, loose fixings, etc prior to use. If any of the components of the instrument are found to be of suspect condition, take remedial action before assembling and using the instrument. Of particular importance are the safety critical components such as the laser safety windows and electrical lid interlock of the sample chamber. Seek advice from the manufacturer if necessary. Do not operate LIBSCAN with any of the covers removed.

Step 2 Prior to connecting the laser power supply, personal computer etc, the LIBSCAN head should first be placed on a suitable surface such as a laboratory bench or optical table. It is highly desirable to secure the LIBSCAN head in some way (eg. by utilizing the feet attached to the base of the LIBSCAN head and which are designed to suit a metric or imperial optical table) to prevent accidental dropping of the head resulting in possible damage to the sensitive components inside.

Step 3 If the sample chamber is to be used, fit the LIBSCAN head to the sample chamber using the four M5 screws (supplied with the unit) as illustrated in the previous figures.

Step 4 Connect the 12 Volt DC (2.5 Amp) plug-in mains power adaptor to the spectrometer console.

Step 5 Connect the coolant pipes and electrical cables (supplied with the laser) between the LIBS head and the ICE 450 laser power supply. Refer to the laser manufacturer's instructions.

Step 6 Using the supplied interlock cable, connect the "Interlock IN/OUT" port on the spectrometer console to the Interlock Override Unit located on the top front of the ICE 450 laser power supply.

Step 7 If there is a need to use an external laser warning light (eg. located at the entrance door to a laser laboratory), this should be connected to the "Warning Light" port on the spectrometer console. The LIBSCAN system is supplied with a spare cable plug for this purpose. The "Warning Light" port provides a 15 VDC (150 mA max.) voltage when the laser is in an active state (ie. when the flashlamp ready switch on the laser remote control box is activated). The 15 VDC is derived from the ICE 450 laser power supply via the "Interlock IN/OUT" lead. A protective fuse (150 mA) is fitted to the right of the "Warning Light" port and should only be replaced with the same rating of fuse.

Step 8 Using the supplied BNC-to-BNC cable, connect the "Trigger In" port on the spectrometer console to the "Q-Switch Out" port on the front of the laser power supply.

Step 9 If the gas-purge feature is to be used, connect a suitable inert gas supply (Argon, Helium, Nitrogen, Air) to the "Inert Gas Supply" port using 4 mm OD flexible tubing (a length of this type of tubing is supplied with the LIBS instrument – coloured green). **WARNING - the gas supply MUST be externally regulated to restrict pressure to less than 5 psi (2 to 3 psi should be adequate) and to control the flow-rate. DO NOT USE FLAMMABLE GASES!**

Step 10 Follow instructions supplied with laser for correct procedure for adding coolant (deionised) water to the laser power supply.

Step 11 Open laser safety shutter (located on LHS side panel of LIBSCAN head) by turning handle to horizontal position.

Step 12 Activate laser by switching on key switch (see instructions supplied with laser).

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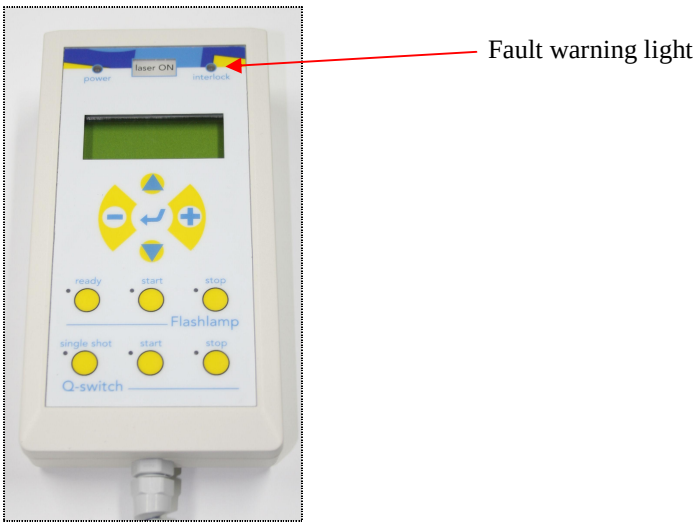
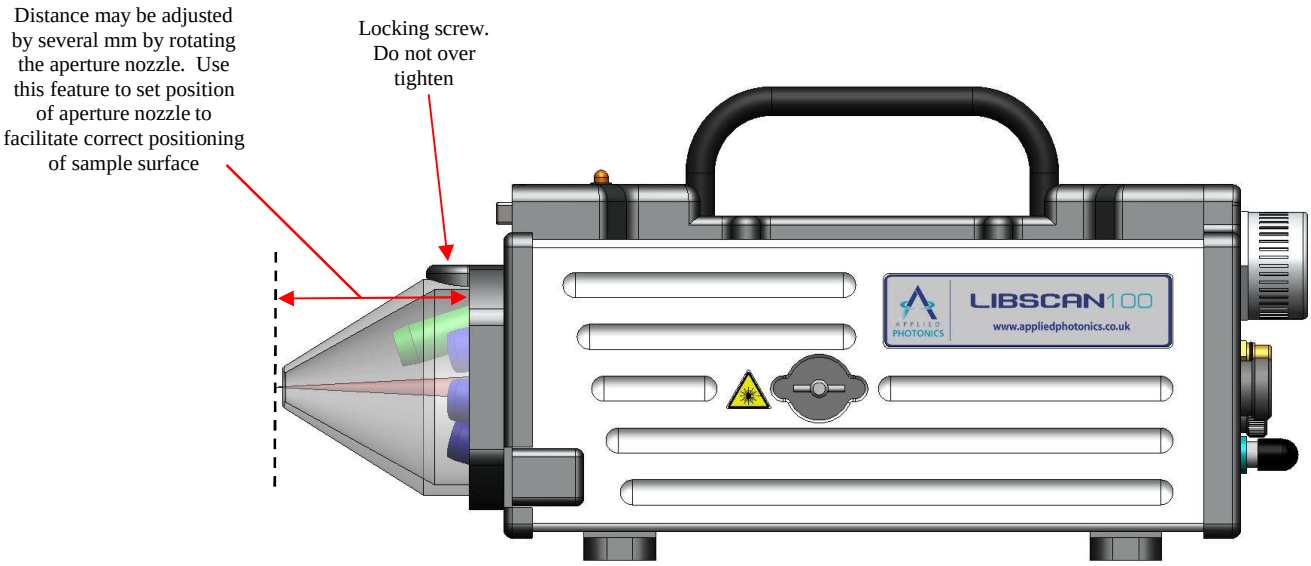
Step 13 With the laser switched on (coolant water flowing, but laser flashlamp not yet activated), check for correct operation of the safety interlock by observing the "Interlock" light on the front panel of the laser power supply controller (see following figure) and opening / closing the door of the sample chamber (with LIBSCAN head fitted and all interlock cables correctly fitted). If the interlock is working correctly, the "Interlock" light should flash when the sample chamber lid is open and be constantly illuminated when the lid is closed. If the interlock is found not to be operating correctly, refer to the *Fault Finding* section of this User's Manual. Note that if the LIBSCAN head is removed from the sample chamber, the interlock circuit is designed to de-activate the laser (see section 6 on *Operating instrument in "open-beam" configuration*). **Warning – do not operate LIBSCAN if the safety interlock is not functioning correctly**.

Step 14 Place a sample of material (eg. a metal block) at the focal plane of the Nd:YAG laser beam inside the sample chamber. Close the door of the sample chamber. Using the controls on the Remote Box of the ICE 450 laser power supply (see following figure and refer to laser manufacturer's instructions), the laser beam may now be fired by i) first activating the flashlamp and ii) then activating the Q-Switch. If the sample material is located at or near to the focal plane of the laser beam, a laser-induced plasma will be produced on the surface of the sample. It may be necessary to adjust the position of the aperture nozzle to obtain correct positioning of the sample surface (see figure below).



View of front panel of laser power supply controller (referred to as Remote Box in Quantel's documentation)


Distance may be adjusted by several mm by rotating the aperture nozzle. Use this feature to set position of aperture nozzle to facilitate correct positioning of sample surface
Locking screw. Do not over tighten
LIBSCAN100
www.appliedphotonics.co.uk

CAD view of LIBSCAN head showing method for adjusting position of aperture nozzle

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Step 15 Connect the personal computer to the "Computer I/O" port of the spectrometer console using the supplied USB 2.0 cable.

Step 16 Connect the personal computer to the serial port on the front panel of the ICE 450 laser power supply using the supplied Serial-to-Serial lead. If the personal computer is not equipped with a serial port, it will be necessary to use a Serial-to-USB converter (note that this device usually requires the installation of a software driver on the personal computer – refer to instructions written on the CD:ROM supplied with the Serial-to-USB converter lead).

Step 17 Follow the instructions provided with the LIBSoft software to correctly configure the spectrometers and the laser for acquiring data. Note that the laser Remote Box should not be used while LIBSoft is running as this will result in the laser reverting to manual control. To resume LIBSoft software control of the laser, it will be necessary to switch off the laser and close LIBSoft, then switch on the laser followed by re-starting LIBSoft.

Step 18 Adjustment of the laser output energy is achieved by adjusting the Flashlamp-to-Q-Switch (FL-QS) delay time. When running the laser in manual mode, the FL-QS delay time may be adjusted via the Remote Box (refer to Quantel's instructions for the laser). The factory setting is typically around 140 microseconds for maximum laser output energy, although this will vary from laser to laser. Increasing the FL-QS delay will reduce the laser output energy. The user will only be able to adjust the FL-QS delay setting from the minimum factory setting (typically 140 microseconds) up to 500 microseconds, although the laser output energy will likely be approaching zero with a FL-QS delay setting of approx. 400 microsconds. When running the laser via LIBSoft, the laser energy may be adjusted in a similar fashion but by entering an appropriate FL-QS delay time via LIBSoft rather than the Remote Box.

Step 19 After successfully testing LIBSCAN, it is now ready for use. Measurement conditions such as Nd:YAG laser pulse energy and position of sample surface with respect to laser beam focal plane will need to be adjusted to suit the requirements of the experiment.

5. Shut-down procedure

Step 1 Shut down LIBSoft software (if being used) and then switch off laser power supply and isolate from mains electrical supply.

Step 2 Close the laser safety shutter on the LIBSCAN head by turning the handle (located on LHS side panel of LIBSCAN head) to vertical position.

Step 3 Isolate 12 Volt DC plug-in power adaptor from mains electrical supply and disconnect from spectrometer console.

Step 4 Disconnect gas supply if connected.

6. Operating LIBSCAN in "open beam" configuration

Warning Class 4 Laser Product.
Only suitably trained and experienced persons under the supervision of a duly appointed Laser Safety Officer should operate LIBSCAN in "open beam" configuration.

LIBSCAN may be used without the sample chamber if required. Under these conditions, the laser beam is not contained (ie. the equipment is a Class 4 laser product) and hence additional safety precautions must be observed including the use of appropriate laser protective eyewear and operating LIBSCAN in a

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suitable controlled environment (eg. a laser laboratory). **Important - it is the responsibility of the user to conduct an appropriate risk assessment prior to using LIBSCAN in open-beam mode**. If the sample chamber is not fitted to the LIBSCAN head, it will be necessary to activate the Interlock Override Unit by turning the key switch to the "ON" position (ie. turn clockwise). The two LED warning lights will commence flashing (assuming the laser is in an active state).

WARNING: As outlined in Section 2.1, activating the Interlock Override Unit will disable the sample chamber safety interlock feature and allow the LIBSCAN head to emit potentially hazardous levels of laser radiation (i.e. Class 4 laser product). It is the responsibility of the user to ensure that safety is maintained when operating LIBSCAN in this configuration. As a minimum, it will be necessary to operate LIBSCAN in a laser controlled area (e.g. a laser laboratory) and for all personnel in the controlled area to wear appropriate laser protective eyewear. Consult your Laser Safety Officer before operating LIBSCAN in this mode.

7. Maintenance and inspection

LIBSCAN should be periodically inspected for signs of damage or wear and tear. Of particular importance are the safety features including the laser safety windows and the laser safety interlock mechanisms and associated electrical circuits. If any damage to the laser safety windows is observed or suspected, or the sample chamber door interlock switch is not functioning correctly, LIBSCAN should be temporarily removed from service until the fault is rectified. For maintaining and inspecting the Quantel Big Sky laser, the documentation supplied with the laser should be consulted. If in any doubt, contact the manufacturer, Applied Photonics Ltd, for further advice on maintenance and inspection of the product.

8. Shipping and storage

The LIBSCAN system should be kept in a clean, dry environment which is free from extremes of temperature. The equipment contains sensitive optical and electro-optical components and so should be protected from excessive vibration or shock. During transport, the LIBSCAN system and ssociated components should be packed in such a way as to prevent damage from shock or vibration and protected from ingress of dust. For shipping or storage of the laser, refer to the laser manufacturer's instructions for the correct procedure.

The laser power supply and laser head (within the LIBSCAN head) MUST be drained of coolant water if there is any possibility of the unit being exposed to temperatures below 4 Celsius (eg. during transit). Failure to do so could result in serious damage to the laser head and/or the laser power supply. The laser manufacturer's instructions should be followed for draining coolant water from the laser power supply and the laser head.

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9. Trouble-shooting / fault finding

Laser does not activate:

1. Is the laser shutter on the LIBSCAN head set to the OPEN position?
2. Is the safety interlock connected correctly?
3. Is the "Interlock" indicator light on laser power supply controller flashing? If yes, then check that sample chamber door is fully closed. If a sample chamber is not fitted to the LIBSCAN head and the equipment is required to be operated in "open-beam" (ie. Class 4 Laser Product) mode, then the Interlock Override Unit will need to be activated.
4. If the safety interlock appears to be working correctly but the laser still does not activate, refer to the operating instructions provided with the laser.

Laser-induced plasma appears to be adequately intense although a poor signal is observed on some or all of the spectrometer channels:

1. Are the data acquisition settings on the LIBSoft software set up correctly?
2. Is the composition of the sample such that emission lines are not expected to be seen on some or all of the spectrometer channels? If yes, then use an alternative sample which has numerous emission lines (eg. an iron-containing material such as steel) to check for correct operation of the LIBSCAN system.
3. Is the sample positioned correctly relative to the laser beam focus? It may be necessary to adjust the position of the aperture nozzle.
4. Is the laser pulse energy too low? Increase if necessary.

Recorded spectra show some emission lines which are saturating the detector ("flat top" appearance to the emission line):

1. Reduce the pulse energy of the laser using the controls on the front panel of the laser power supply (manual mode of operation) or via the LIBSoft software (software control mode of operation).
2. Increase spot size of laser beam on sample by adjusting the position of the sample relative to the focal plane of the laser beam.

Recorded spectra show some emission lines suffering from "self-reversal" (ie. a "dip" in the centre of the emission line):

1. Reduce the pulse energy of the laser.
2. Increase spot size of laser beam on sample by adjusting the position of the sample surface relative to the focal plane of the laser beam (it is usual to set focus to be approximately 1 to 3 millimetres "into" the sample surface)

Air-breakdown is observed in the path of the laser beam just in front of the sample surface:

1. The focal plane of the laser beam is set incorrectly (ie. it is set to be in front of the sample) – adjust position of sample so that the focal plane of the laser beam is coincident, or preferably just "into", the sample surface.
2. On irradiation by the laser beam, the sample is creating considerable quantities of particulates in the path of the laser beam. Try cleaning the surface of the sample if loose material (eg oxide, surface contamination etc) is present. Reducing the laser energy and/or increasing laser beam spot size on the surface of the sample may also help.

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Appendix A1

Quick guide to acquiring spectra using LIBSoft V1.0

1. Start the LIBSCAN system

- Ensure that all cables are connected correctly (refer to schematic diagram on page 19 of this User's Manual)
- Apply 12 VDC power to the LIBSCAN console
- Power-up the laser
- Power-up the system computer
- Start the LIBSoft application
- Check that the LIBSoft recognises the laser and, if software control of the spectrometer(s) is required, all of the spectrometers are recognised. Note that the current version of LIBSoft only supports Avantes spectrometers – contact Applied Photonics Ltd for further information on this.

2. Configure the laser

- Select ***Configuration>Configure laser*** from the menu or click on the blue ***LASER*** shortcut button to open the laser configuration window
- Set the Q-switch ***Delay (us)*** parameter to the required value
  This parameter adjusts the laser energy – the lower the value the higher the energy
  NB The default value when the laser is first powered-up is the minimum value of Q-switch delay for the specific laser in use, i.e., the maximum energy
- Set the Q-switch ***Rep rate (Hz)*** parameter to the required value
  2 Hz is a typical value
  Occasionally errors may be generated at high rep rates (ie. > 5 Hz) due to time taken to acquire data from the spectrometers following each Q-switch trigger pulse and the acquisition may then time out. If this proves problematic reduce the rep rate to the next lower value
- Click the ***OK*** button to save the laser parameter settings and close the laser configuration window

3. Configure the spectrometers

- Select ***Configuration>Configure spectrometers*** from the menu or click on the blue ***SPECT*** shortcut button to open the spectrometer configuration window
- Set ***Start wavelength*** and ***Stop wavelength*** parameters for each spectrometer so that there is no wavelength overlap between spectrometers
  For example, if the *Stop wavelength* of one spectrometer is 353.1 nm the *Start wavelength* of the neighbouring spectrometer should be set to 353.2 nm
  Once set, these values are stored by the system, but can be changed at a later date if required
- Select which spectrometer is to be used as the ***Master***
  This is the spectrometer to which the Q-switch trigger from the laser is connected
  The Q-switch trigger output is connected to the spectrometer with the highest wavelength range by default
  Once set, this value is stored by the system, but can be changed at a later date if required
- Ensure that each of the required spectrometers is ***Activated***
  If activated, the ***Status*** of the spectrometer will be identified as ***IN_USE_BY_APPLICATION***
- Set the ***Integration Time (ms)*** parameter to the required value
  Recommended value = 1.1 ms
- Set the ***Integration Delay (us)*** parameter to the required value
  Recommended value = 1.27 us
- Ensure that the ***Mode*** parameter is set to ***Ext Trig***

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§ Click the ***OK*** button to save the spectrometer parameter settings and close the spectrometer configuration window

4. Configure the measurement parameters

§ Select ***Configuration>Configure measurement*** from the menu or click on the blue ***MEAS*** shortcut button to open the measurement configuration window

§ Set the ***Number of Measurements*** parameter to the required value

§ Set the ***Number of Spectra*** parameter to the required value

§ Set the ***Number of Accumulations*** parameter to the required value

§ Set the ***Number of Conditioning Shots*** parameter to the required value

NB A measurement is defined as the acquisition of data from one target location on a sample. One measurement consists of the specified *Number of Spectra* each of which comprises the specified *Number of Accumulations* at that same target location. The specified *Number of Conditioning Shots* are fired prior to the acquisition of the first spectrum of a measurement in order to condition the sample surface. For a *Number of Measurements* > 1 the sample must be moved between measurements.

§ Set the ***Sample name*** parameter to the required value

§ Set the ***Sample filepath*** parameter to the required value

§ Set the ***Background name*** parameter to the required value
Only required if background correction is enabled

§ Set the ***Background filepath*** parameter to the required value
Only required if background correction is enabled

§ Enable/disable the ***Auto-increment filename*** function as required
If enabled, a numeric extension starting at *Current value* and incrementing by 1 for each subsequent measurement will be added to the *Sample name* to form the filename under which measurement data will be saved

§ Enable/disable the ***Auto-save spectra*** function as required
If enabled, the acquired spectra will be displayed and automatically saved
If disabled, the acquired spectra will be displayed and the user will be given the opportunity to save or discard the data

§ Enable/disable the ***Background correction*** function as required
It is suggested that this function is enabled
If enabled, the background spectrum specified by *Background name* and *Background filepath* is subtracted from each acquired spectrum
If enabled, it is important to ensure that a background spectrum has been acquired using the ***same*** spectrometer and measurement parameters as to be used for the measurements before any measurements are made

§ Click the ***OK*** button to save the measurement parameter settings and close the measurement configuration window

5. Acquire background spectrum

NB This is only required if the Background correction function has been enabled and a suitable background spectrum does not already exist

§ Select ***Acquisition>Acquire background*** from the menu or click on the orange ***B/G*** shortcut button
A spectrum will be acquired and displayed using the Master spectrometer's internal trigger to initiate the acquisition and a file dialogue box will be displayed allowing the user to save the spectrum

§ Save this background spectrum
NB The spectrum can be saved using the default filename (the *Background name* set on the measurement configuration screen) or a new name. If a new name is used the *Background name* on the measurement

configuration screen will be updated accordingly and this file will be used to correct subsequent measurement data

6. Acquire measurement spectra

- Position the sample in front of the laser aperture
- Select ***Acquisition>Acquire spectra*** from the menu or click on the green ***ACQ*** shortcut button

  The laser flashlamps will be started if not already on and the laser Q-switch will fire in accordance with the specified laser and measurement parameters
  The acquired spectrum will be displayed and, if the *Auto-save spectra* function is enabled, saved.
  On completion of the measurement acquisition the laser Q-switch will be stopped, but the flashlamps will remain on
  If more than one measurement has been specified, the user will be prompted to move the sample to the next target location
  The spectrum can be viewed in more detail by maximising the spectrum display window and/or using the zoom and cursor function buttons
  If the *Number of spectra* > 1, each spectrum in the measurement can be viewed individually by adjusting the *Spectrum Number* control on the spectrum display window
  If the *Auto-save spectra* function is disabled, the displayed spectrum data can be saved by selecting the *Save* option from the menu on the spectrum display window
  To stop a measurement while the system is acquiring click on the red *STOP* shortcut button

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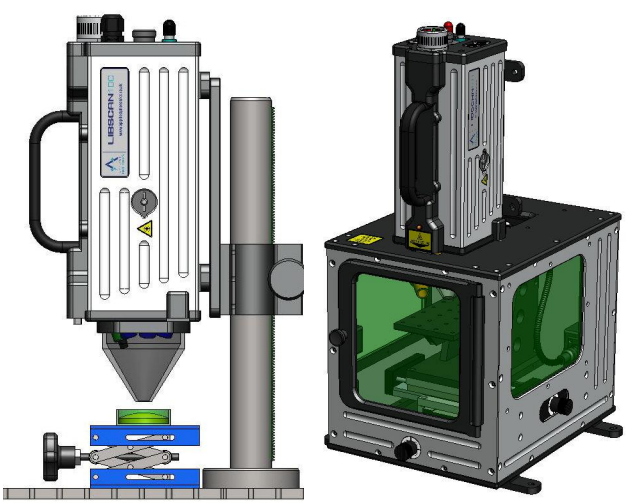
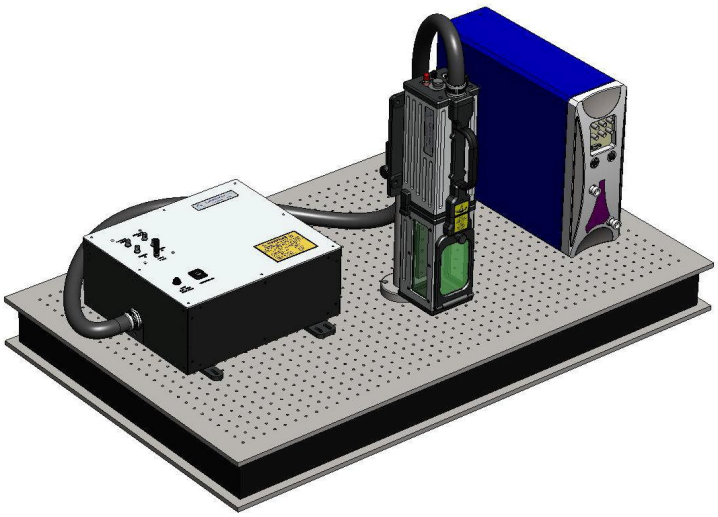
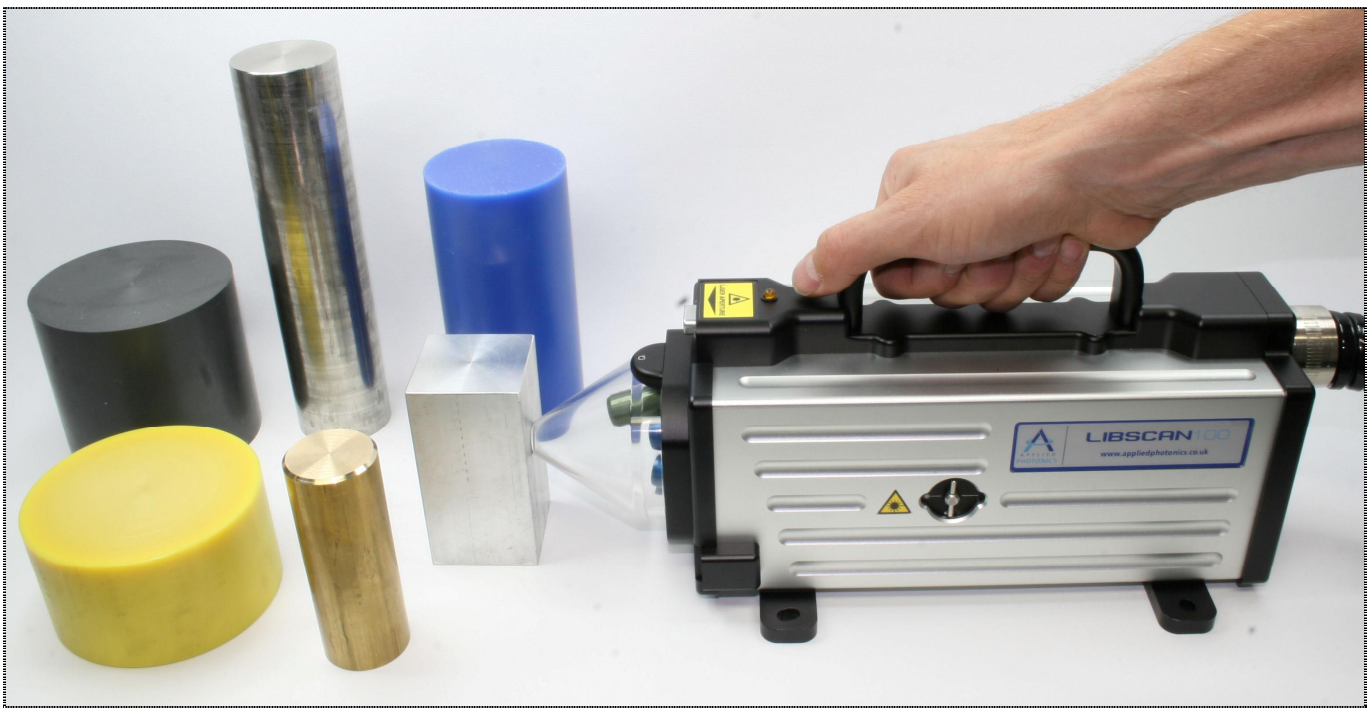
Appendix A2

Example configurations of LIBSCAN system







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Appendix A3

Certificate of Conformity

Applied Photonics Limited
Unit 8 Carleton Business Park
Skipton North Yorkshire
BD23 2DE United Kingdom

EC
Declaration of Conformity

Applied Photonics Ltd declares that the product listed below has been designed and manufactured in compliance with the relevant standards as follows:

Product name: **LIBSCAN Modular LIBS System**

Model Number: **LIBSCAN 50 & LIBSCAN 100**

Laser product safety
This device conforms with the principal objectives of safety of laser products by application of the following standards: **PD IEC TR 60825-14:2004 and BS EN 207:1999**

Electrical Safety
This device conforms with the principal safety objectives of the **European Directive 73/23/EEC**, as implemented by the Electrical Equipment (Safety) Regulations 1994, by application of the following standard: **BS EN 61010-1:2001.**

Electro-Magnetic Compatibility
This device conforms with the principal objectives of the European Directive (89/336/EEC) as amended by 91/31/EEC and 93/68/EEC, as implemented by The EMC Regulations (SI 1992 No. 2372 and amendment SI 1994 No. 3080), by application of the following standard: **BS EN 61326-1:2006**

Year of affixation of the CE Marking: 2010

Signed:

Name: Andrew I. Whitehouse

Title: Managing Director

Place: Applied Photonics Ltd, Unit 8 Carleton Business Park, Skipton, North Yorkshire BD23 2DE, United Kingdom

Date: September 2010

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